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AFWAL-TR-87-2036

PRODUCTION OF HIGH ENERGY AVIATION FUELS FROM ADVANCED COAL LIQUIDS

JOHN DOWNEN

STRAT CO. 4597 JUPITER DRIVE SALT LAKE CITY, UTAH 84124

**APRIL 1987** 

FINAL REPORT FOR THE PERIOD JUNE 1986 - FEBRUARY 1987

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This technical report has been reviewed and is approved for publication.

WILLIAM E. HARRISON III

Fuels Branch

Fuels and Lubrication Division Aero Propulsion Laboratory ARTHUR V. CHURCHILL, Chief

Fuels Branch

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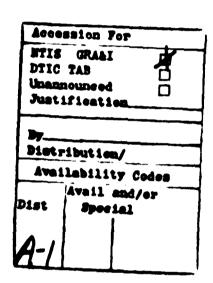
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LETTER AND REPORT FROM J & A ASSOCIATES, INC
APPENDIX C





### PRODUCTION OF HIGH ENERGY AVIATION FUELS

### from

### ADVANCED COAL LIQUIDS

John Downen, Principal Investigator

Strat Co., 4597 Jupiter Drive, Salt Lake City, Utah

March 15, 1987

### Introduction and Acknowledgments

Strat Co. initiated an innovative research effort supported by the AFWAL/Aero Propulsion Laboratory to examine the feasibility of producing higher energy aviation fuels from domestic coal resources. This effort would address two issues; first, it would assess aspects of a domestic resource, coal liquids, that have heretofore been considered to be of little value as an aviation fuel feedstock; and, secondly, it would produce liquids suitable as fuels for aircraft of increased speed and range.

The work was conducted at the Fuels Branch of the Aero Propulsion Laboratories at Wright-Patterson Air Force Base. The assistance of the staff of the Fuels Branch is gratefully acknowledged, for its conduct of the experimental and analytical work of the project.

Conventional aviation fuels are comprised primarily of normal and iso-paraffins which are the major constituents of most crude oils. Aromatic molecules naturally exist in some crude oils and others are generated in refinery cracking and reforming processes. Olefins are almost exclusively a result of the cracking and coking process. Aromatic molecules are significantly more dense than paraffinic molecules and therefore contain substantially more energy per unit volume. Unfortunately, aromatics do not burn well in conventional engines and as a consequence maximum aromatics specifications and corresponding minimum smoke point specifications have been established for conventional jet fuels.

Naphthenic molecules appear to be the best prospects for improved hydrocarbon aircraft fuels. They retain most of the advantages of both paraffins and aromatics. Like paraffins, the naphthenes are saturated and thus are more stable than the unsaturated aromatics and olefins. Naphthenes also burn more like paraffins, without most of the smoke problems related to aromatics. Because naphthenes are cyclic paraffins, their density is greater than the normal and iso paraffins that make up most of the volume of

conventional fuels. Naphthenes therefore contain more energy per unit volume than conventional fuels.

Unfortunately, naphthenes do not naturally occur in most crudes in high enough concentrations to significantly affect the energy density of conventional aviation fuels. The most likely processes for making the volumes of naphthenes needed for advanced fuels will involve careful hydroprocessing of aromatic molecules of the appropriate size in order to saturate their double bonds without breaking their rings, thus converting them to naphthenes. Hydrocarbon liquids derived from certain coals contain high percentages of those aromatic molecules and may become one of the prime sources of the feedstocks needed for production of advanced fuels.

Coal tars as secondary products from coal gasification projects were originally considered as possible feedstocks for this program. As a first approximation, the characteristics of these materials were found to be not suitable for volume production of high quality fuels. The difficulties related to their processing, transport and storage and the potential carcinogenicity of the heavier tars were the principal factors in this decision. These materials also are now not expected to become widely available on the domestic market in the near future. For a number of reasons, therefore, a middle distillate of the coal liquids from the DOE/EPRI Advanced Two-Stage Liquifaction Facility (TSL) which is operated by Catalytic Inc. at Wilsonville, Alabama was determined to be a better feedstock for Phase I of this program.

The feedstock evaluated in this effort was derived from upper seam Wyodak (Wyoming subbituminous) coal which was processed at the TSL. It was produced in Run #251 during August 1986 under the sponsorship of the Department of Energy, the Electric Power Research Institute (EPRI) and several industrial supporters. A schematic of the TSL facility is included in Appendix B as is a report on the composition of the liquid before it was fractionated.

Two drums of the TSL liquids were fractionated by J&A Associates, Inc. of Golden, Colorado in a 25 gallon batch still. Only the 340°F to 590°F cut (ASTM D86) was shipped to AFWAL for hydrotreatment into candidate fuels (see Table 2). Appendix B also includes a letter report from J&A outlining the results of the distillation of the two drums along with pertinent data.

Sullivan 1,2,3 had previously evaluated coal liquids from the TSL and produced fuels which met current jet fuel specifications. In this effort, the objective was not necessarily to produce specification fuels but to produce a variety of liquids which might be considered for future advanced applications. Because of a lack of product fractionation equipment and of fuel "polishing" facilities such as clay treatment and additives, no real attempt was made to produce finished specification fuels.

The effect of reaction conditions on the quality of fuel was determined within the limitations of the analysis equipment at AFWAL. In addition, this effort was Phase I of a more comprehensive program which would optimize fuel production conditions and evaluate these materials in simulated advanced propulsion applications. In the Phase II expansion of this effort, feedstocks other than coal liquids, including energetic gases, would also be evaluated and compared. Some details of the Phase II effort will be further discussed in the "Recommendations" Section of this report.

### The Hydrogenation Reaction System

The reaction system used in this program was located in the Fuels and Lubrication Building (Building 490) in Area B at Wright-Patterson Air Force Base near Dayton, Ohio. The Hydrogenation Reaction System (HRS) was an automated pilot plant capable of studying the processing associated with feedstocks such as petroleum, heavy oils, tars, coal liquids, shale oil etc. Its main function, however, is to study hydrogenation and hydrocracking processes needed to produce suitable candidates for transportation fuels such as jet and diesel from a variety of sources. The unit was built by Xytel Inc. of Chicago from a design based on Air Force requirements.

Appendix B includes a schematic of the HRS. It is basically a two-reactor system with the capability of operating the reactors in series. For instance, in upgrading high fuel-bound nitrogen feed like western shale oil, the first reactor could be used as a "guard" reactor to remove nitrogen to the parts per million range, remove metals such as arsenic and iron and to saturate olefinic hydrocarbons, to keep the main hydrocracking reactor from being poisoned. This procedure is analogous to typical refinery operations with these materials and is used in the Unocal shale oil upgrading facility near Parachute, Colorado. However, in this study only one reactor was used since "life studies" on the catalyst were beyond the scope of the effort.

The HRS facility has the normal capability of supplying liquid and gaseous fluids to the reactors. The temperatures in the reactor are monitored and controlled by a thermocouple located in a thermowell on the centerline of the reactor. The thermocouple can traverse the length of the reactor. The facility has the capability of liquid feed rates up to 600 cc/hr with hydrogen flows being limited to approximately 35 standard cubic feet per hour. The reaction temperatures are limited to approximately 950°F with a pressure capability of nearly 3000psig. The reaction products can be scrubbed to remove hydrogen sulfide and ammonia and the gaseous products can be separated from the liquids. Gas analysis using an on-stream gas chromatograph can be made to facilitate accurate material balances. The facility is physically located in Room 154 of Building 490 in a high-bay

area equipped with more than adequate safety precautions. A complete description of the HRS is expected to be available in reports originating from the AFWAL Fuels and Lubrication Division and will not be presented here.

This facility will be of great value to the USAF as it develops its understanding of the processes for producing high quality fuels for future hypersonic aircraft. In addition the basic system is being modified so it can be configured to evaluate catalyst systems for endothermic fuels needed for high speed flight in the atmosphere. The operations made possible by this configuration will become the focus of the Phase II part of this program.

### Test Results

A series of hydrogenation tests were performed on the coal liquid (340°-590°F) distillate to evaluate the effect of the reaction parameters on the resulting products. Sufficient products were obtained to provide analytical data to ascertain the quality of the products as suitable feedstock for candidate fuels. Complete jet fuel specifications on the products were not run since no attempt was made to produce "polished" usable fuels. Such tests as corrosivity and thermal stability could be met through correct finishing processes such as stripping and clay treatment. The object of this study was to produce suitable material for further refining including desired fractionation.

No attempt was made to evaluate catalyst life. Before a valid economic analysis of the cost of the products could be made, data on the life of the catalysts would have to be obtained by further work, as it has a major effect on refinery economics and must be maximized for production operations. Further work would also be needed in order to develop information for projections for scale-up and continuous production of any of these liquids. This was outside the scope of the Phase I study and will be developed from the Phase II data.

For this effort two catalysts were selected to represent conventional off-the-shelf hydrotreating catalysts. They were a nickle-molybdenum (NiMo) and a precious metal catalyst which usually has increased hydrogenation activity. Shell 424, NiMo on alumina, was tested on the feedstock in 85% of the runs and this data was compared to an Engelhard sulfided 0.5 wt% Pt on carbon catalyst which was acquired from the Hercules Research Center at Wilmington, DE. A descriptive brochure on the Shell 424 catalyst is included in Appendix B.

Reaction conditions studied included temperatures:  $600^{\circ}-700^{\circ}$  F, pressures: 2000-2500 psig, and feedrates: 0.19-0.39 lbm/hr (LHSV: 0.5 to 1.0). Table 1 is the test matrix as the project was conducted. Further tests were scheduled with the Pt catalyst and another series was planned with a nickle-zinc catalyst but

strong symptoms of an imminent failure of the main hydrogen pump of the HRS ended the test series before they were run.

A summary description of the feedstock is presented as Table 2. Appendix B includes the reports from J&A associates and from Catalytic Inc. that describe it in more detail. Appendix B also includes a diagram of the reactor and a discussion of how it was loaded. Appendix A is a suite of graphic presentations of the test data.

The products were analyzed for density, weight percents of sulfur, nitrogen and hydrogen and for chemical types by GC/MS for certain key products. The data resulting from the sulfur analysis is anomalous. A moderate drop in sulfur concentration was observed as the feedstock was hydrotreated. However, under the severity of the operating conditions, it was expected that the sulfur would be essentially removed during hydrotreating. In spite of this problem, the data from the sulfur analyses are presented in this report. It is likely that the actual concentration of sulfur in the product is near zero. A more appropriate method of analysis for low sulfur concentrations will be used in Phase II.

A complete description of reaction conditions is included as Appendix C together with all test results except chromatographs and peak identification data.

Tables 3-8 describe the effects of reaction conditions on the products from the tests performed in this project. This effort, (SBIR Phase I), was to be a scoping study to indicate directionally where Phase II should be initiated to gain fuller insight into advanced fuel production from the point of view of reaction conditions and economics.

Most of the reactions studied were carried out over the Shell 424 catalyst. The effect of reaction temperature at 2000psig is presented in Table 3. The products produced at 600°-700°F are compared to the feedstock. At a LHSV of 1 and hydrogen feed of 12,100 scf/bbl, sulfur and nitrogen contents were reduced while the hydrogen content of the products increased with temperature. The density of the products decreased (increased API gravity) due primarily to the hydrogenation. Very little cracking occurred at these conditions but probably would have become significant at higher reactor temperatures and lower pressures. The apparent sulfur reduction with temperature was not dramatic (from 400 to 30 Uppm) and is almost certainly indicative of imprecise analytical techniques. The feedstock sulfur content (438ppm) is higher than the J&A analyses (131ppm) indicated. This anomaly will be cleared up in Phase II with a better understanding of these analytical methods.

Nitrogen levels were reduced from 3000ppm to 20-60ppm at the test temperatures; these figures are probably more accurate than the sulfur assays. Assuming that the data in Table 3 is accurate,

then the Shell 424 catalyst is not effective in producing a sulfur-free or nitrogen-free product for advanced fuel applications where these components would cause high temperature instabilities, although further work and better analyses may lead to conditions of better performance by the catalyst. In any event, the Shell 424 catalyst could be used in guard reactors to reduce nitrogen levels and perhaps trace metal and olefin contents to levels where extremely active catalysts would not be poisoned in downstream hydrogenation/hydrocracking operations.

In Table 4 the effect of reaction pressure on hydrogenation was evaluated in several of the tests. The effect of increased pressure on the reduction of sulfur and nitrogen and the increase in hydrogen content are not apparent from tests run at 2000psig and 2500psig. Tests at lower and higher pressures were beyond the scope of this effort and probably would have given more insight into the effect of greater pressure range. The tests in Table 4 were all run at 700°F, LHSV=1 and using the Shell 424 catalyst. From this minimal data there would seem to be no need to hydrotreat at higher pressures.

The effect of residence time was evaluated and presented in Table 5. These tests were performed at LHSV of 1 and 0.5, at a pressure of 2500psig and temperature of 700°F using the Shell 424 catalyst. Here again, sulfur content of the product was apparently not greatly influenced by the greater residence time (lower flow rate). The nitrogen content was reduced from 74ppm to 50ppm at the lower flow rate and the hydrogen content increased by 2% to 12.93%

Table 6 evaluates the effect of the Shell 424 catalyst compared with the presulfided Engelhard 0.5 wt% Pt on carbon and clearly indicates, with the minimum data obtained, that the Pt is not as effective as the NiMo in removing sulfur and nitrogen from the feed. Also the hydrogen content of the Pt catalyst-produced product was lower than that with the Shell 424 material. These bracketed tests were run at reaction temperature of 600°F, pressure of 2000psig and LHSV=1.

The effect of a higher reaction temperature,  $700^{\circ}\text{F}$ , was demonstrated on the two catalyst types in Table 7. The same results are found as those observed at the lower reaction temperatures. At  $700^{\circ}\text{F}$  the Pt catalyst also produced a much higher gravity (41.70° API) indicating that some cracking may have been occurring. It is highly likely that such trace compounds as sulfur, nitrogen and metals may have poisoned the usually highly reactive Pt catalyst.

In Phase II the Pt catalyst will be used to evaluate advanced fuel production but an appropriate guard reactor will be used. As Pt catalysts are expected to function well in an endothermic regime, the data from the Pt catalyst in this program may serve as baseline information for the next phase. In Phase I, this

approach was not practical based on scheduling and funding limitations.

Results on the two catalyst types at lower feedrates are presented in Table 8 and the results and conclusions are similar to those in Table 7.

### Test Data

TABLE 1

### TEST MATRIX

test no.	$\delta_{\mathbf{F}}^{emp}.$	catalyst type	press. psig	feedrate lbs mass/hr	LHSV	gas rate scfh **
HRS-Ø16	FEEDS	TOCK				
HRS-017	6ØØ	NI-MO	2000	Ø.39	1	15
HRS-Ø18	6ØØ	NI-MO	2000	Ø.39	1	15
HRS-Ø19	6ØØ	NI-MO	2000	Ø.39	1	15
HRS-020	65Ø	NI-MO	2000	Ø.39	1	15
HRS-Ø21	695	NI-MO	2000	Ø.39	1	15
HRS-Ø22	FEEDS	TOCK				
HRS-Ø23	700	NI-MO	2000	Ø.39	1	15
HRS-Ø24	7ØØ	NI-MO	2000	Ø.39	1	15
HRS-Ø25	7ØØ	NI-MO	25ØØ	Ø.39	1	15
HRS-Ø26	7ØØ	NI-MO	25ØØ	Ø.39	1	15
HRS-Ø27	7ØØ	NI-MO	25ØØ	Ø.39	1	15
HRS-Ø28	7ØØ	NI-MO	25ØØ	Ø.19	Ø.5	15
HRS-Ø29	7ØØ	NI-MO	25ØØ	Ø.39	1	15
HRS-030	7ØØ	NI-MO	25ØØ	Ø.19	Ø.5	15
HRS-Ø31	600	NI-MO	2000	Ø.39	1	15
HRS-Ø32	6ØØ	NI-MO	2000	Ø.39	1	15
HRS-Ø33	6ØØ	NI-MO	2000	Ø.39	1	15
HRS-Ø34	6ØØ	Pt	2000	Ø.39	1	15
HRS-Ø35	700	Pt	2000	Ø.39	1	15
HRS-Ø36	700	Pt	2000	Ø.19	Ø.5	15

<sup>\*\*</sup> NOTE: This converts to 12,096 (12,100) scf/bbl of feedstock at LHSV of 1, and 24,200 scf/bbl at LHSV of 0.5, on a one pass through basis (no hydrogen is recycled).

TABLE 2

# DATA ON COAL LIQUID FEEDSTOCK (From J & A Associates Report)

Quantity: 61.3 gallons (at 60 degrees F) Volume per unit of liquid: 58.4

86.83
12.84
Ø.25
Ø.Ø1
$\circ_{\mathbf{F}}$
340
382
437
486
524
566
59Ø

### TABLE 3

### EFFECT OF REACTION TEMPERATURE ON PRODUCTS

Catalyst: Shell 424(Ni/Mo) Feed Rate: Ø.39 lbm/hr Pressure: 2000 psig H2 Feed: 12,100 scf/bbl

LHSV: 1

### Analyses

Reaction Temperature	Sulfur wgt	• .	Hydrogen wgt %	Gravity API					
feed	Ø.Ø44	Ø.3ØØ	12.01	27.45					
600	0.041	Ø.Ø7Ø .	12.44	29.77					
65Ø	Ø.Ø33	Ø.ØØ2	12.93	31.61					
695		0.006	12.76	31.27					
700	0.034	0.006	12.78	31.27					

### TABLE 4

### EFFECT OF PRESSURE ON PRODUCTS

	Catalyst:	Shell	424	Feed	Rate:	Ø.39	lbm/hr
_	_	0			_		

Temperature: 700° F LHSV: 1

### Analyses

Reactor Pressure psig	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	Ø.3ØØ	12.01	27.45
2000	Ø.Ø34	0.006	12.78	31.27
25ØØ	Ø.Ø36	0.007	12.73	31.24

### TABLE 5

### EFFECT OF RESIDENCE TIME ON PRODUCTS

Temperature: 700°F Catalyst: Shell 424

Pressure: 2500 psig

### Analyses

Feed Rate lbm/hr (LHSV)	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	Ø.Ø44	0.300	12.01	27.45
Ø.39 (1)	Ø.Ø32	0.0074	12.70	31.14
Ø.19 (Ø.5)	Ø.Ø29	0.0050	12.93	32.03

### TABLE 6

### EFFECT OF CATALYST TYPE ON PRODUCTS

Pressure: 2000 psig Temperature: 600°F

Feedrate: Ø.39 lbm/hr

LHSV:

### Analyses

	~-~			
Catalyst Type	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	Ø.3ØØ	12.01	27.45
Shell 424 (Ni/Mo)	0.041	0.070	12.44	29.77
Englehard (0.5 Pt		Ø.262	12.29	28.53

TABLE 7 EFFECT OF CATALYST TYPE ON PRODUCTS

2000 psig 700° F Pressure: Temperature:

Ø.39 1bm/hr Peedrate:

LHSV :

Ana	1	v	£	e	8
UI ICI	٠	7	•	•	•

Catalyst Type	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API		
feed	0.044	0.300	12.01	27.45		
Shell 424 (Ni/Mo)	Ø.Ø34	Ø.ØØ6	12.78	31.27		
Englehard (Ø.5%Pt)	Ø.Ø38	0.161	12.57	41.78		

### TABLE 8

### EFFECT OF CATALYST TYPE ON PRODUCTS

Pressure: 2000 psig Temperature: 700° F Feedrate: Ø.19 lbm/hr

> LHSV: Ø.5

### Analyses

Catalyst Type	Sulfur wgt %	Nitrogen wgt %	Hydrogen wgt %	Gravity API
feed	0.044	0.300	12.01	27.45
Shell 424 (Ni/Mo)	Ø.029	0.005	12.93	32.03
Engelhard (0.5% pt	)0.040	0.151	12.49	29.74

### Results of Further Analytical Work

For selected hydrotreating tests, gas chromatographic/mass spectographic analyses were run. This data is useful in identifying basic hydrocarbon types. Saturated hydrocarbons are either straight-chained normal and iso paraffins or mono-and dicycloparaffins. These types of structures represent the highest hydrogen content possible, with n-paraffins having the highest possible content for a given carbon number. The most stable unsaturated hydrocarbons are aromatics, here represented by alkylbenzenes, indans and tetralins, and naphthalenes. These materials are deficient of hydrogen atoms but are stable due to resonance within the aromatic ring. The cyclic compounds can be hydrogenated to cyclic paraffins, i.e. naphthalenes become dicycloparaffins with the saturation of all carbon atoms.

In this effort, the GC/MS data were obtained on selected products and compared to the feedstock. This data is presented in Table 9. Seven classes of compounds are quantified. The first three categories are paraffinic (completely saturated) while the last four are unsaturated aromatics and olefins. Olefins are unsaturated hydrocarbons which are not stabilized by resonance, are usually identified as being unstable and are characterized as gum formers, depending on type. Di-olefins are usually the most unstable.

The coal liquid from Wilsonville was an excellent potential refinery feedstock. It was hydrogenated to a fairly high degree in its production as its hydrogen content was 12.81 wt% with only 25.3% aromatics and 1.2 wt% olefins. This data is summarized by general group (paraffins, naphthenes, aromatics and olefins). Table 18 shows the effect of reaction conditions on these hydrocarbon types. Using the Shell 424 catalyst and reaction pressure of 2000 psig and liquid feedrate of 0.39 lb (mass) / hour (LHSV=1) the effect of increasing reaction temperature is presented. The paraffin content dropped slightly from 16.1% to 13-14% as the temperature was increased. The naphthenes, which are the candidate compounds for use as high density fuels, increased with temperature commensurate with the decrease of aromatics. Also, as would be expected, the total hydrogen content of the product increased as the aromatics and olefins became saturated. As its hydrogen content approaches 13%, a fuel becomes more acceptable for contemporary jet engines. Future engines will benefit from the increased volumetric energy by being designed to operate efficiently on lower hydrogen content and heavier cyclic fuels.

The high remaining paraffin content is probably representative of the long chained waxes which are typical of liquids from subbituminous coal, which have higher freeze points and which would tend to raise the freeze point of the products. The freeze points of these products are significantly higher than those of most conventional fuels. Not much cracking occurred under these test conditions. The freeze points could be decreased by dewaxing or isomerization of the products although a less expensive procedure may be just to decrease the end point by fractionation. Another option would be to use a higher rank coal as the liquifier feedstock which would produce a higher percentage of aromatics and less paraffins. The fractionation facilities were not operable at the Air Force laboratory for Phase I but should be available in Phase II to facilitate production of as broad a range as possible of suitable materials. Process economics will be compared based on using wider cuts and converting the waxes to lower melting isomers versus fractionating a more narrow boiling range product.

Cyclohydrocarbons are more dense than the corresponding paraffins, see Table 11 where n-decane is compared to decahydronaphthalene (decalin). Both molecules contain ten carbon atoms. The specific gravity of the straight chained n-

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decame is 0.7342 compared to 0.9011 for the cis-decalin. Also the dicyclic decalin produces a lower freeze point and an 18% increase in volumetric heating value. The drawback in using cyclic hydrocarbons is the reduction in hydrogen content which affects fuel combustibility in terms of increased flame radiation and soot formation. These shortcomings can be dealt with and to a considerable extent overcome by engine design.

Table 9

### GC/MS RESULTS ON SELECTED PRODUCTS

Test Number, HRS-(See Table 1 For Corresponding HRS Test Operating Conditions)

### Hydrocarbon type, wt%

Paraffins	16.1	13.3	13.4	13.2	14.0	12.1	15.0	13.9
Monocycloparaffins	33.4	37.0	38.0	38.0	39.1	39.6	34.9	33.8
Dicycloparaffins	24.0	26.6	33.7	32.2	30.6	35.3	25.3	27.5
Alkylbenzenes	7.1	6.3	5.5	5.9	5.8	5.2	6.4	6.8
Indan & Tetralins	13.9	13.1	7.4	8.6	8.3	6.0	13.4	14.6
Naphthalenes	4.3	3.1	1.5	1.6	1.8	1.1	3.4	3.1
Olefins	1.2	0.7	0.5	Ø.5	0.5	0.6	0.6	0.9

### Table 10

### EFFECT OF REACTION TEMPERATURE ON PRODUCT COMPOSITION

Catalyst: Shell 424
Pressure: 2000 psig
Feed Rate: 0.39 lbm/hr
H2 Feed: 12.100 scf/bbl

	Reac	tion T	emperat	ure, o	e, <sup>o</sup> F			
Hydrocarbon type, wt %	feedstock	600	650	695	700			
Paraffins	16.1	13.3	13.4	13.2	14.0			
Naphthenes	57.4	63.6	71.7	70.2	69.7			
Aromatics	25.3	22.5	14.4	16.1	15.9			
Olefins	1.2	0.7	0.5	0.5	0.5			
Total saturates	73.5	76.9	85.1	83.4	83.7			
Total unsaturates	26.5	23.2	14.9	16.6	16.4			
Hydrogen, wt%	12.01	12.40	12.93	12.76	12.93			

	n-decane	decalin
Formula	C <sub>10 H22</sub>	C <sub>10</sub> H <sub>18</sub>
Molecular Weight	142.29	138.25
Specific Gravity Boiling Point, OF Freeze Point, OF	0.7342	0.9011
Boiling Point, OF	345.5	384.5
Freeze Point, OF	-21.4	-45.6
Characterization Factor, K	12.61	10.50
Hydrogen, Wt%	15.59	13.12
Net Heat Of Combustion		
BTU/lb	19,017	18,324
BTU/gal	116,410	137,660

### Conclusions

Although the US interest in alternative domestic fossil fuel resources is currently in a lull, it is of interest to visualize the potential for producing from them not only high quality conventional fuels but the families of advanced fuels needed for the next generations of aircraft. Future effort in this area must be timely, aggressive, meaningful and technically sound.

Comparisons of characteristics of products made with current fuel specifications indicates that the Strat Co. effort demonstrated, within the constraints of the SBIR Phase I resources, that coal liquids could be processed into quality fuels. The feedstock which was fractionated into a cut suitable for jet fuel production was a high grade material; as received, the concentrations in it of sulfur, nitrogen and oxygen were lower than in most petroleum crudes. This material had been hydrostabilized as part of its production. Liquids from the proper coals processed in this facility undoubtedly represent one of the best sources currently available for feedstocks for experimental high density jet fuels. Developing the concept of endothermic or heat sink fuels and the need for cyclic hydrocarbons, like those available from liquified coal, could provide an impetus not only for initiating a viable domestic coal liquids industry but for producing quality advanced fuel for 21st century fighters, interceptors, cruise missiles and aerospace planes and ultimately for production of refinery feedstocks to supplement conventional crudes.

Using conventional processing technology and limited support, Strat Co. demonstrated that advanced fuels are possible within the state-of-the-art. The Shell 424 catalyst is representative of this technology and produced suitable feedstock for future fuels. More extensive information is needed, however, to fully

define future fuel candidates and areas where more sophisticated processing is needed. Pure or nearly pure hydrocarbon compounds or mixtures of them will probably be needed for certain advanced missions; these materials must be identified and specified and arrangements made for their production in a timely and economical manner.

Future efforts and modifications to the hydrotreating facility should enable the AF to study basic heat effects on fuels to gain insight into the realistic reaction kinetics and potential heat sink capabilities of candidate fuels. This information would then be used to design, build and operate a simulator for completely evaluating the fuel/fuel systems interface under more realistic conditions.

In looking at the applications of this data to further study for the production of advanced fuels from coal liquids, the hydrocarbon species needed for endothermic fuels, high density fuels and the other advanced concepts are most likely to be naphthene molecules that are fully saturated and that don't have the problems associated with fuels containing high percentages of aromatics. From the data it is clear that we have substantially increased the percentage of naphthenes. Most coal liquids are very rich in aromatics and most of the aromatics can be converted to naphthenes by proper hydrotreating. Obviously, most of the double bonds are saturated to form naphthenes although some of the rings are broken and the aromatics in that case are converted to alkanes.

Under the operating conditions of this study, the aromatics percentage of the treated product was higher than for specification fuels. By further variations of the operating parameters and catalyst selection this liquid could certainly be made into specification fuel. In addition, this treatment would also increase the concentration of naphthenes in the liquid.

The relative concentrations of the various hydrocarbon species vary greatly among coals of different origin, quality, and rank. For start material, one would select a high volatile, low-ash, low sulfur coal of medium rank. The details of the operating parameters of the coal-to-liquid conversion unit will also affect the relative concentrations of the hydrocarbon species These processes can typically be varied to affect, within limits, the amount of aromatics produced from any particular coal; hence the selection process for producing any particular advanced fuel should commence with coal selection, followed by coal-to-liquid process selection and optimization of process operating parameters, then proceed through selection of a sequence of processes and corresponding operating parameters to distill, hydrotreat, and further fractionate and concentrate the desired species. Final concentration would probably be accomplished mainly by liquid-liquid extraction techniques.

Initially it would appear that producing an advanced fuel by selecting an optimum coal that is rich in aromatics, producing liquids from it under the proper conditions, hydrotreating the aromatics to naphthenes and concentrating them may furnish advanced fuels at lower cost than making them by synthesis from pure compounds. It also is likely that, although a group of similar naphthenic compounds would be produced rather than single compounds, the desired fuel characteristics that are being determined from work with single compounds could be obtained from such a group.

After hydrotreating there will still be some aromatics and paraffins remaining in the liquids. There are a number of commercial processes available to remove both paraffins and aromatics to arrive at a liquid that is composed entirely of naphthenic compounds of similar molecular weight. Following are representative examples:

For extracting the aromatics, one of a number of commercial liquid-liquid extraction processes could be used. Conceptually, this involves a solvent that dissolves the aromatics but not the paraffins or naphthenes. Commercial processes of this type are sulfolane extractors used to remove benzene, toluene, and xylene from high-octane reformate, NMP (n-methyl pyrrolidine) used to remove aromatics from lube oils, and SO2 extraction used to remove aromatics from jet fuel. These are the obvious processes and there are others that could be adapted to this application.

A number of commercial processes exist for eliminating paraffins. Molecular sieves are available designed so that naphthenes and probably much of the iso-paraffins will pass through the sieve and the paraffins will not, and therefore can be recovered by back-flushing. In addition, there are two common commercial dewaxing catalysts, one licensed by British Petroleum which selectively breaks the long chains into light ends under hydrotreating conditions and lowers the pour point (or freeze point) of the liquid. The other, licensed by Mobil, behaves much like a fluid catalytic cracking unit with a hydrogen atmosphere where the paraffins are generally converted to olefins and aromatics in almost a de-hydrogenation reaction. A third catalytic dewaxing process was developed by Union Oil Co. in the late 1970s for dewaxing hydrotreated shale oil to reduce its pour point so it could be pipelined from Parachute Creek to the Fruita refinery without the necessity for heating the pipeline. was not commercially available as of two or three years ago, but arrangements could probably now be made for it.

### Recommendations

The AF Fuels Branch has prided itself in keeping pace with fuel development activities. This became clear with the leadership role it took in developing fuels from domestic (western) oil shale. However, recent cutbacks in energy research by both industry and government dictate that the AF play a larger role if advanced fuels are ever to be practical. Recent funding cuts and manpower reductions require assessment of the modus operandi of the Laboratory. Research contracts must be well defined and hard hitting; opening up new areas of advancement and not merely "reploughing old turf".

strat Co. plans to assist the AF in this challenging new endeavor. For Phase II, an aggressive, imaginative program will be proposed to assist in upgrading the in-house capability of the AF Fuels Laboratory. Outside consultants from industry and academia will be made available to review the background of fuel requirements and areas of future development. The internal facilities of the AF will be operated in an efficient manner to develop prototype fuels in a timely fashion. Current equipment should be modified to supply basic heat sink and chemical reaction parameters for future designs. Chemical analyses must be improved in both type and precision. Difficulty in obtaining correct data in a timely fashion will not be satisfactory if the AF is to move out in this area.

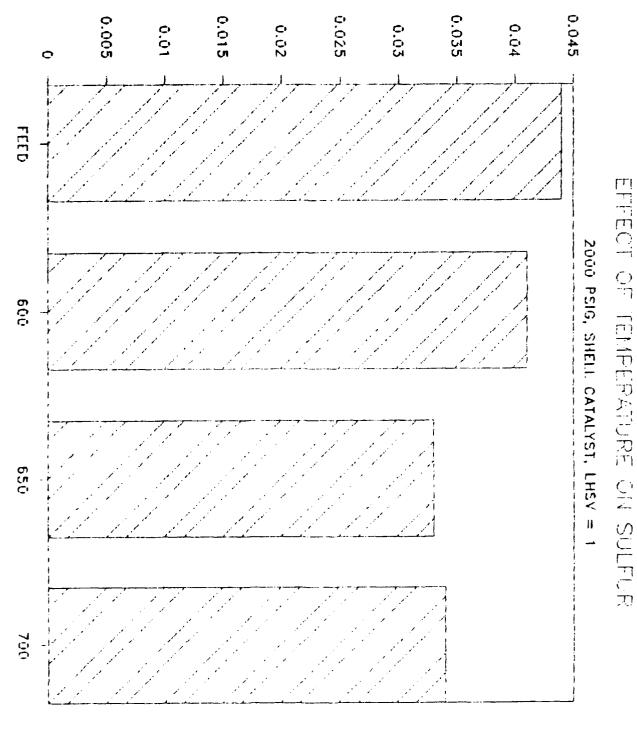
Strat Co. will present a proposal for a Phase II program which will enable the AF to begin to answer the major fuels-related questions posed by Project Forecast II before they disappear by default from lack of attention.

### References

- 1. Richard F. Sullivan, "Two-Stage Catalytic Hydrocracking of ITSL Oil for Jet Fuel and Naphtha", Symposium on Catalytic Processes in Coal Conversion, presented before the Division of Petroleum Chemistry, Inc., American Chemical Society, Chicago Meeting, September 8-13, 1985
- 2. Richard F. Sullivan, "Effects of Feed Boiling Range on Hydrotreating of Wyodak and Illinois ITSL Oils", Paper for DOE Direct Liquefaction Contractor's Conference, Pittsburg, Pennsylvania, November 19-21, 1985
- 3. Richard F. Sullivan and Harry A. Frumkin, "Refining Coal Liquids: Where We Stand" Chevron Research Company, undated
- 4. Coordinating Research Council, Inc. "Handbook of Aviation Fuel Properties" 1983, CRC Report No. 530, CRC, Inc. 219 Perimeter Center Parkway, Atlanta GA 30346

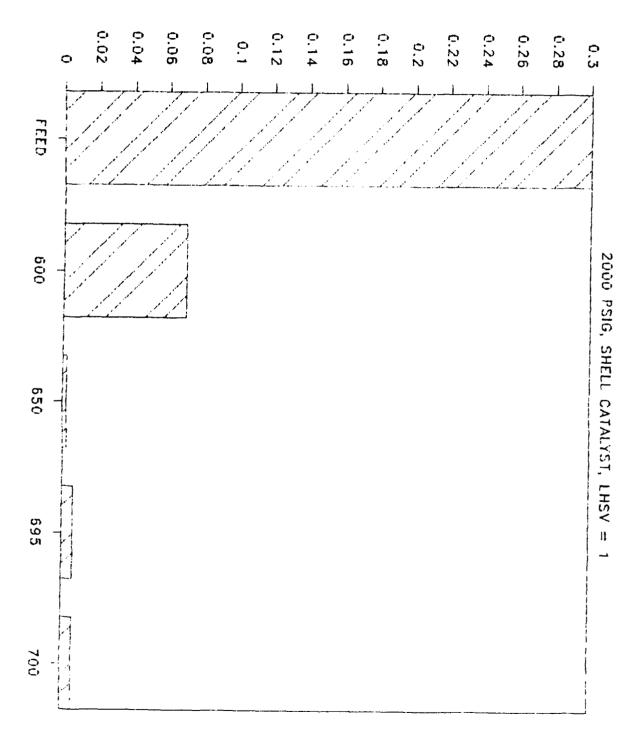
# A P P E N D I X A GRAPHIC PRESENTATION OF TEST DATA

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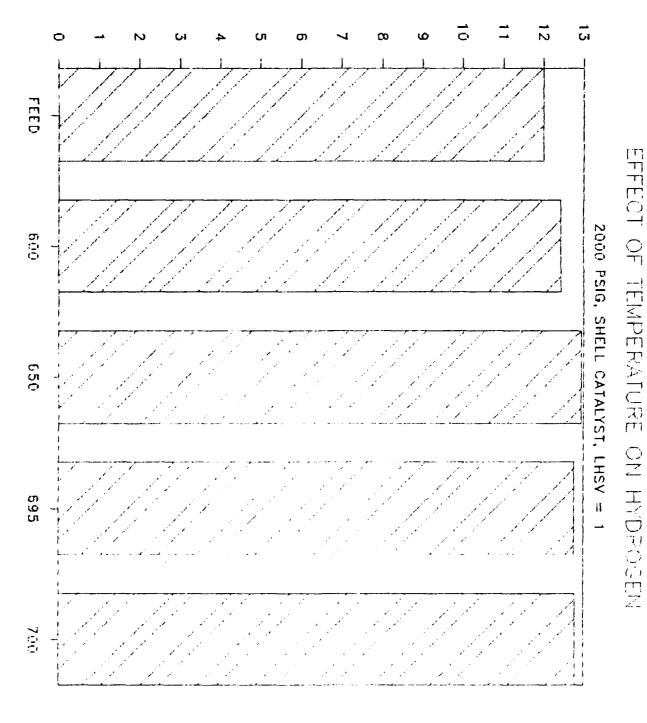
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### WEIGHT & NITROGEN

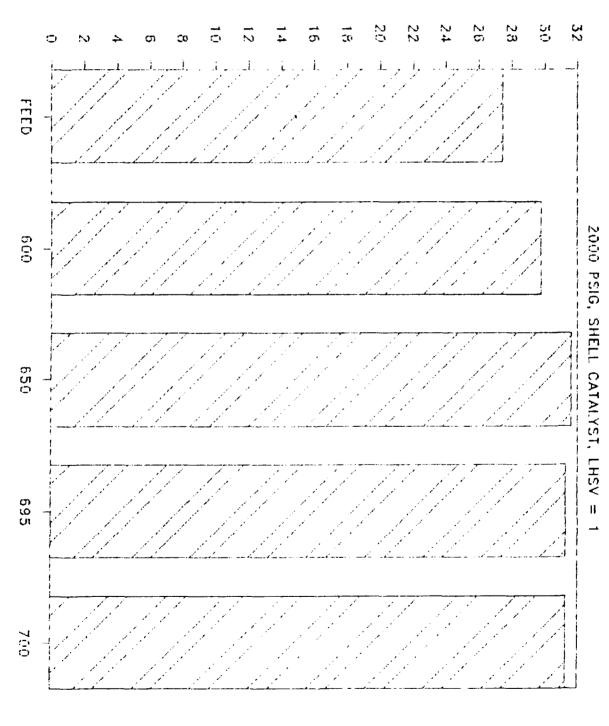


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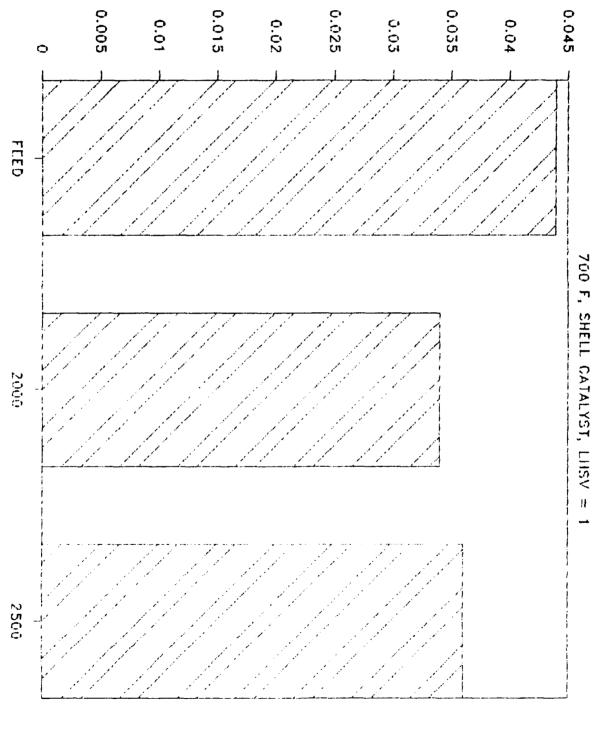
### WEIGHT % HYDROGEN



EFFECT OF TEMPERATURE ON DENSITY

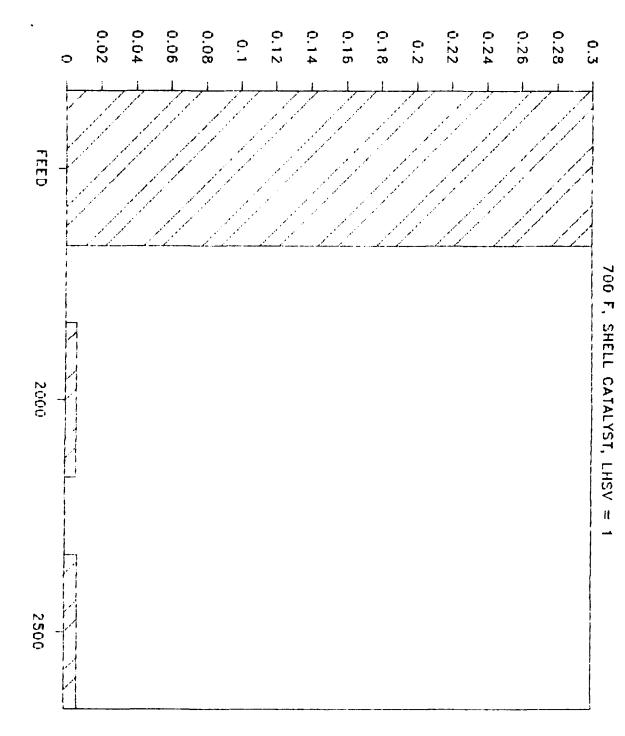


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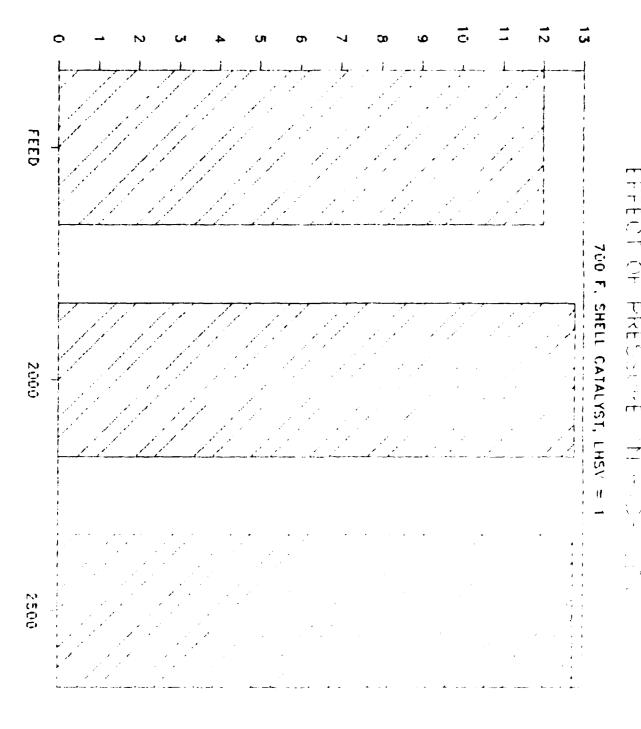


EFFECT OF PRESSURE ON SULFUR

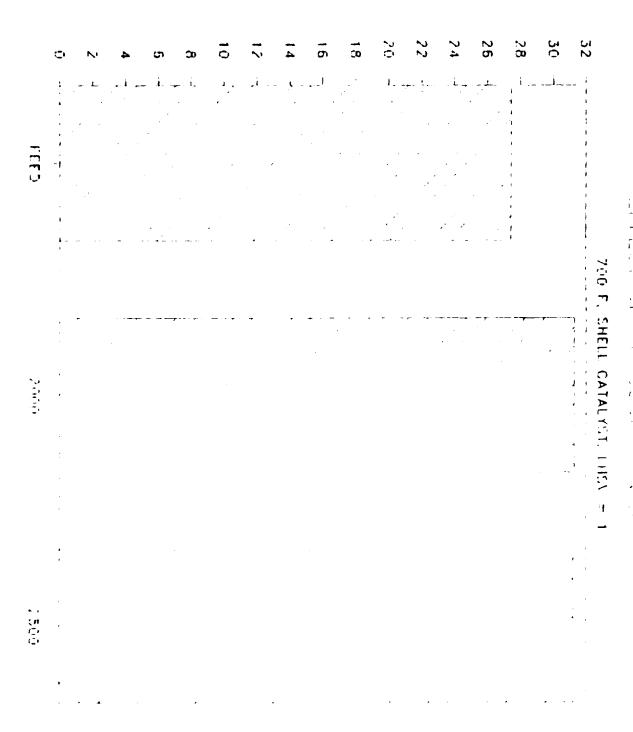
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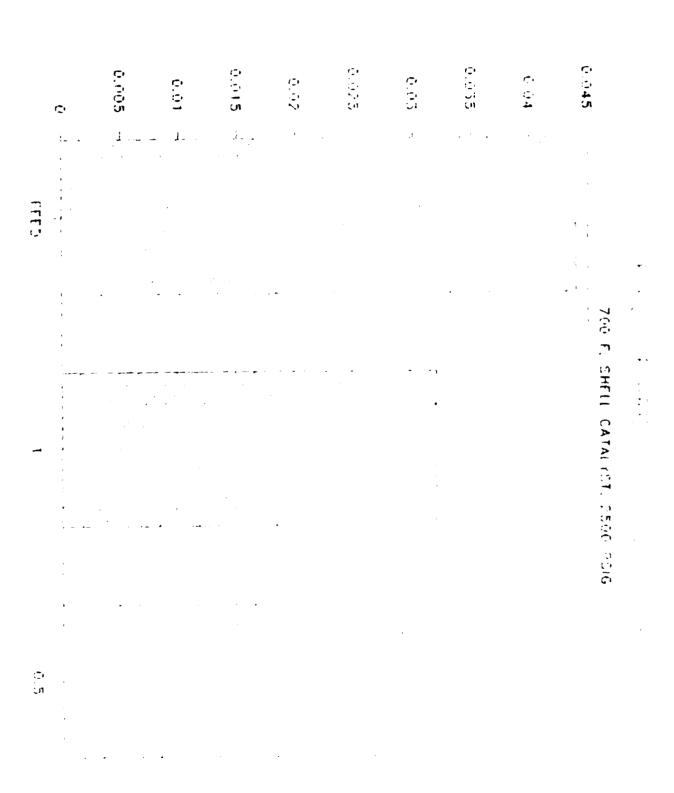
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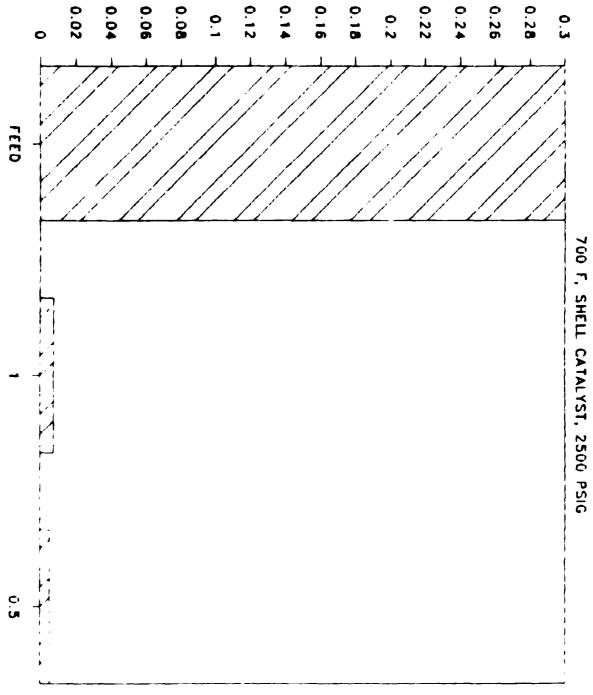


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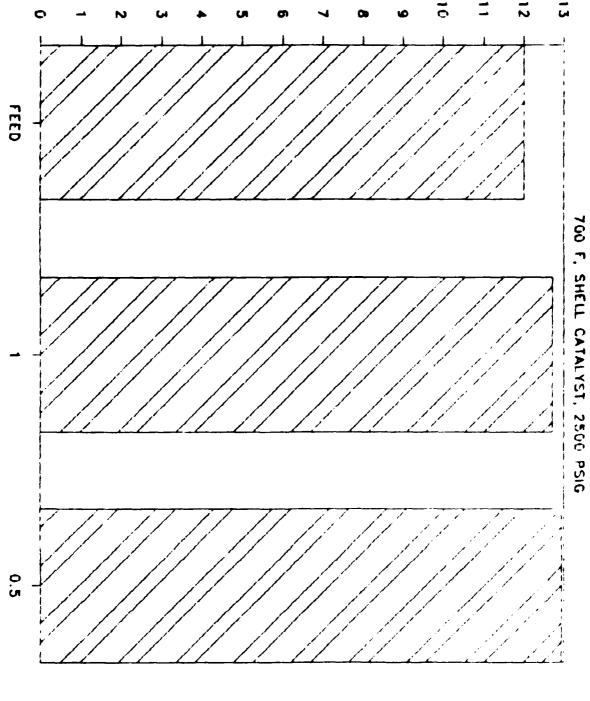
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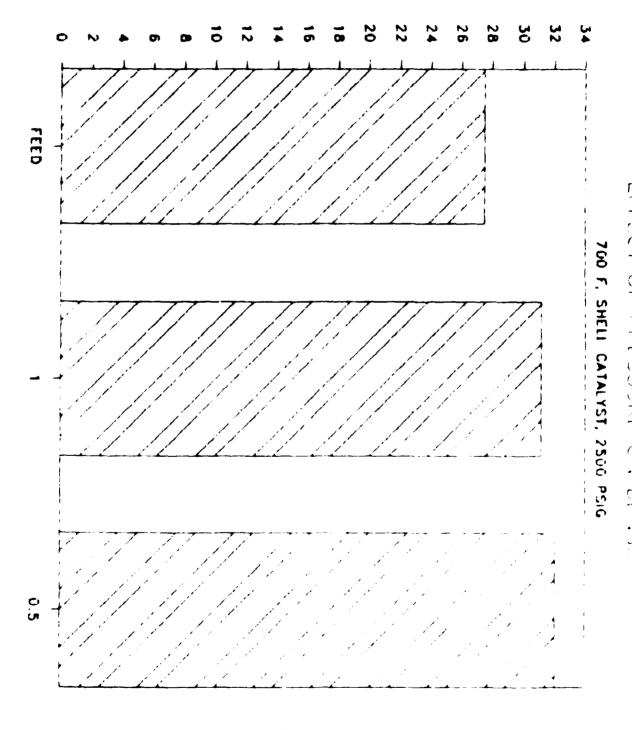
EFFECT OF PRESSURE ON NITROGEN

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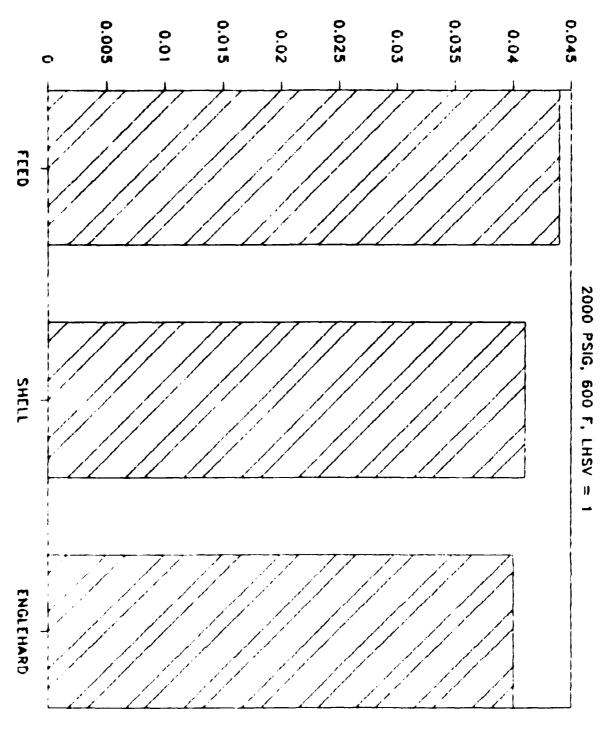
EFFECT OF PRESSURE ON HIDROGEN

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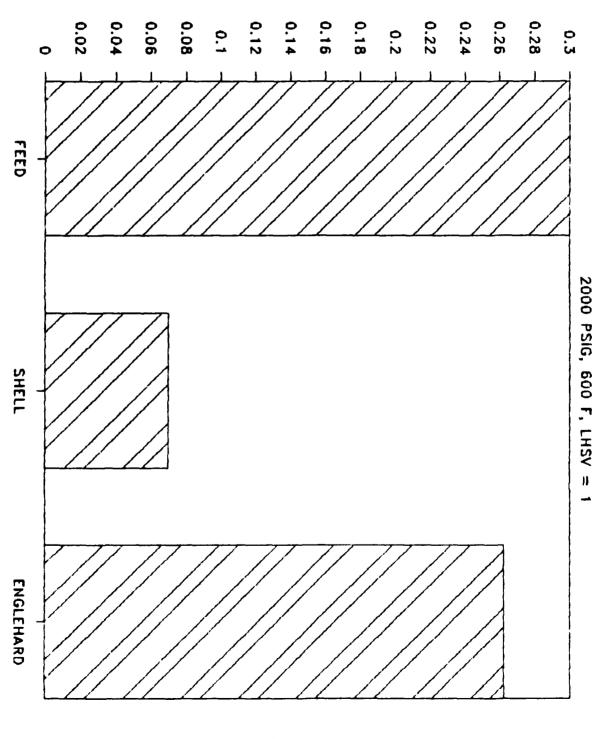
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### WEIGHT \* SULFUR



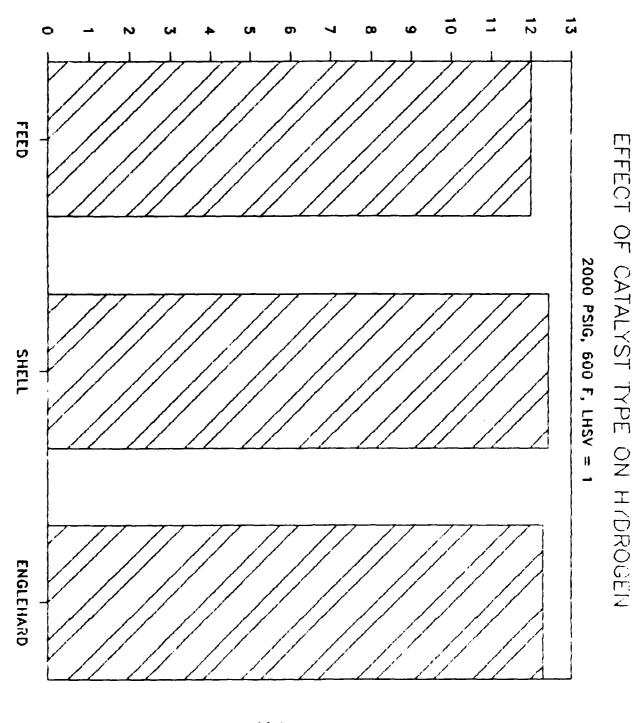
EFFECT OF CATALYST TYPE ON SULFUR

### WEIGHT % NITROGEN

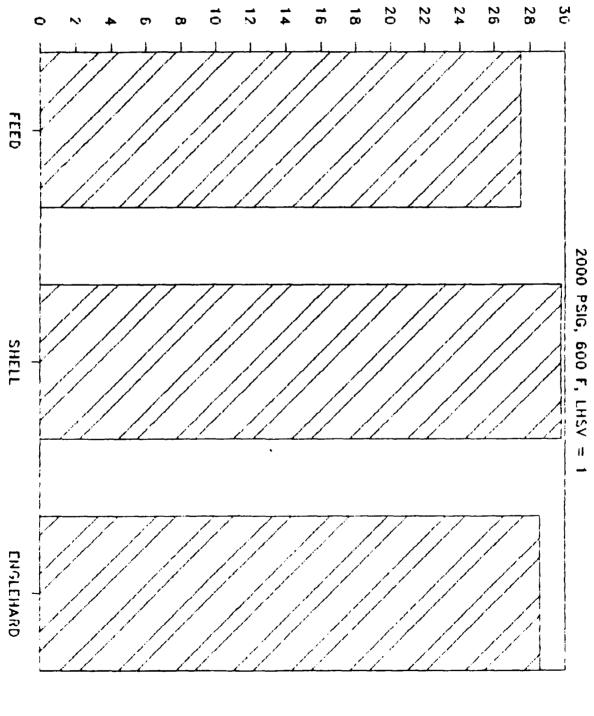


EFFECT OF CATALYST TYPE ON NITROGEN

### WEIGHT % HYDROGEN



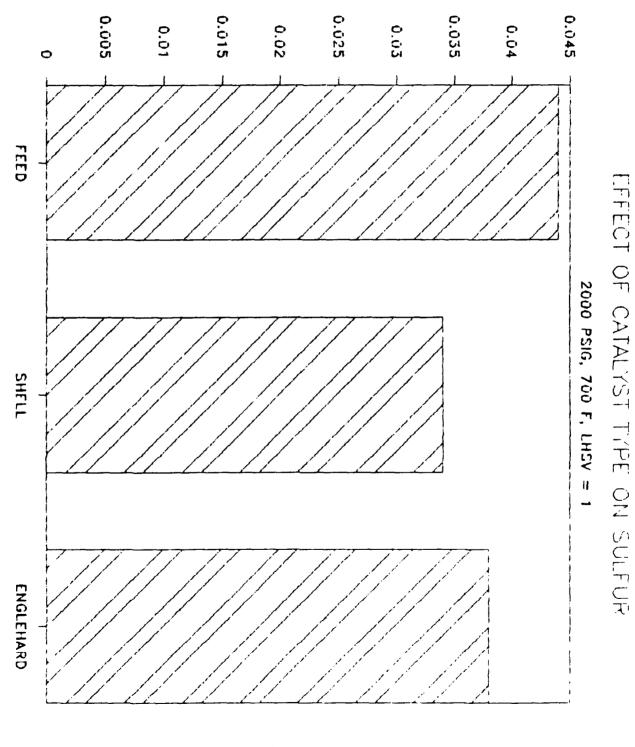
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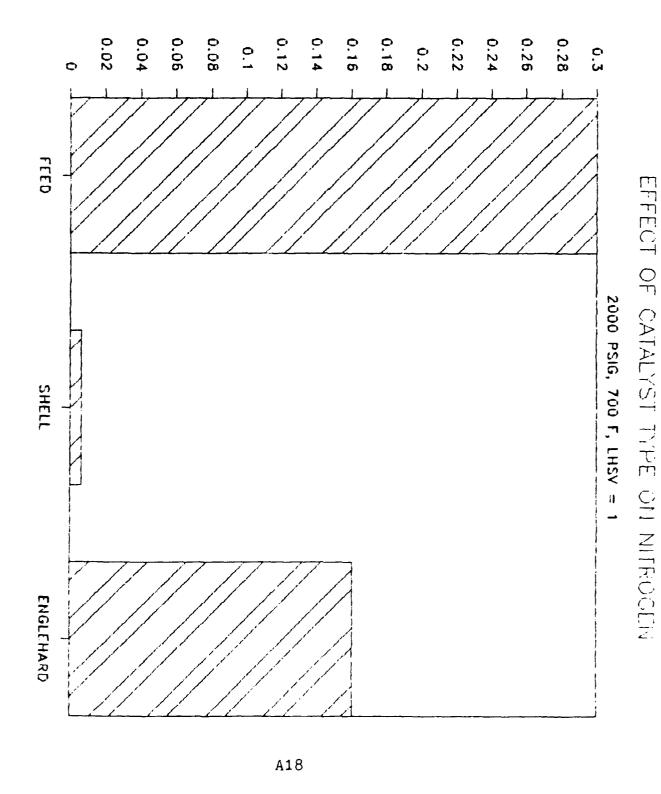
EFFECT OF CATALYST TYPE ON DENSITY

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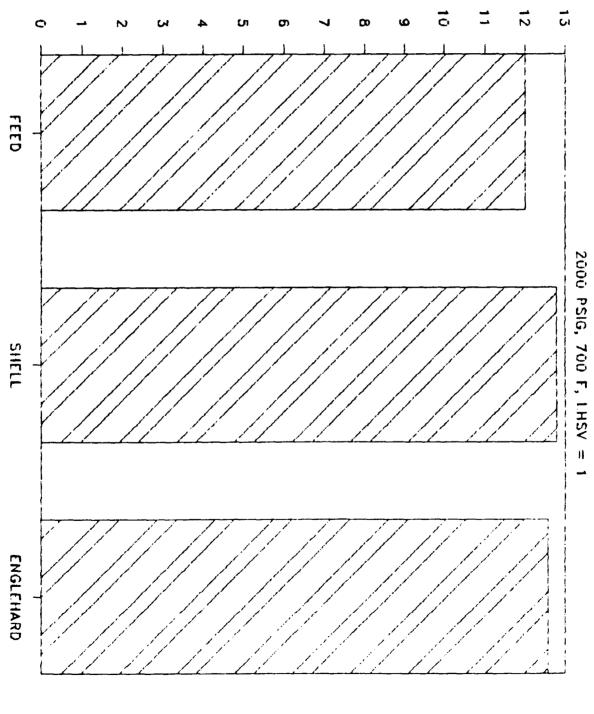
### WEIGHT & SULFUR



### WEIGHT % NITROGEN

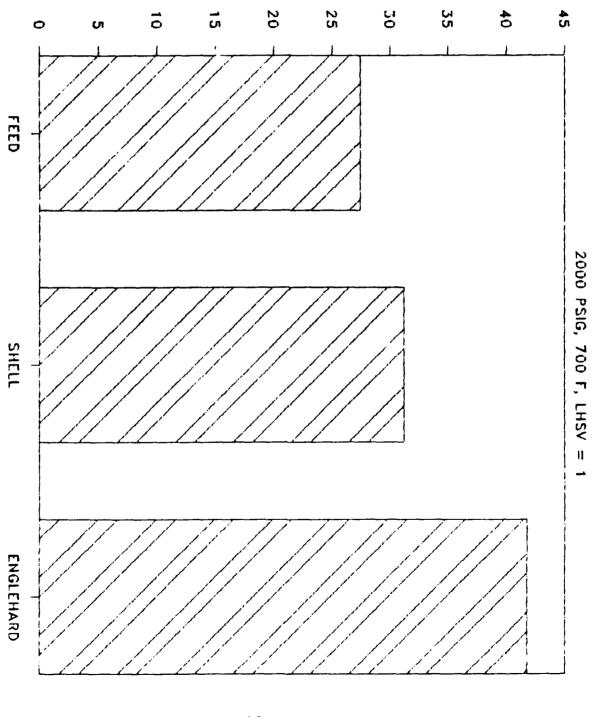


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EFFECT OF CATALYST TYPE ON HIDROGEN

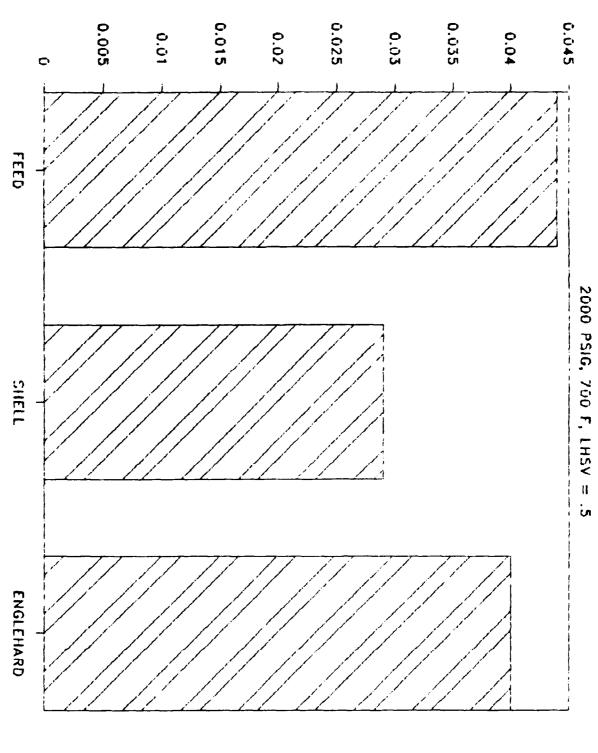
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EFFECT OF CATALYST TYPE ON DENSITY

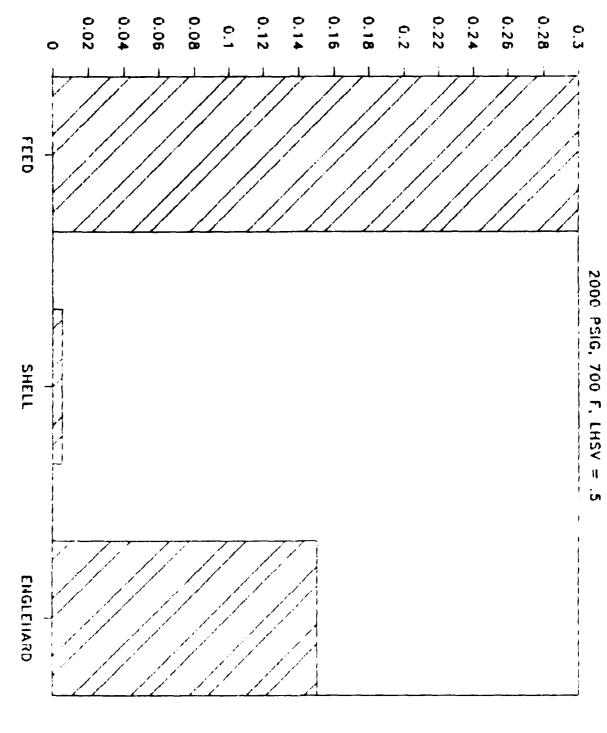
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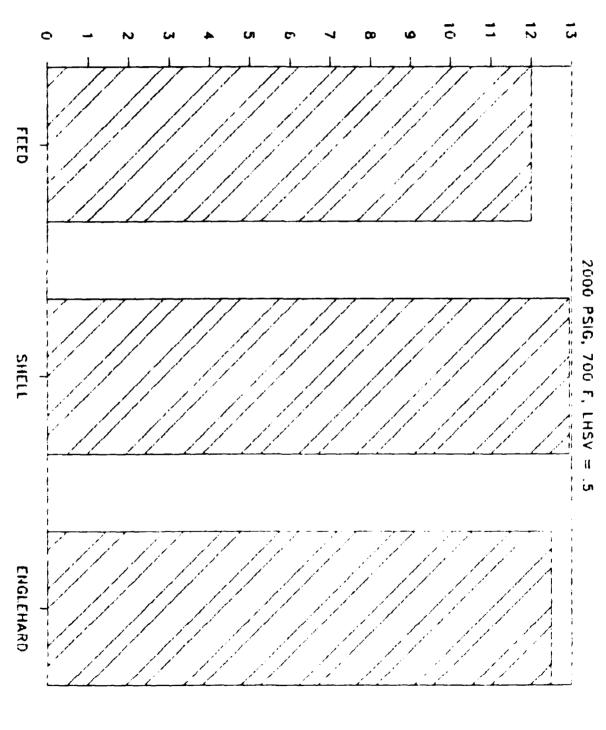


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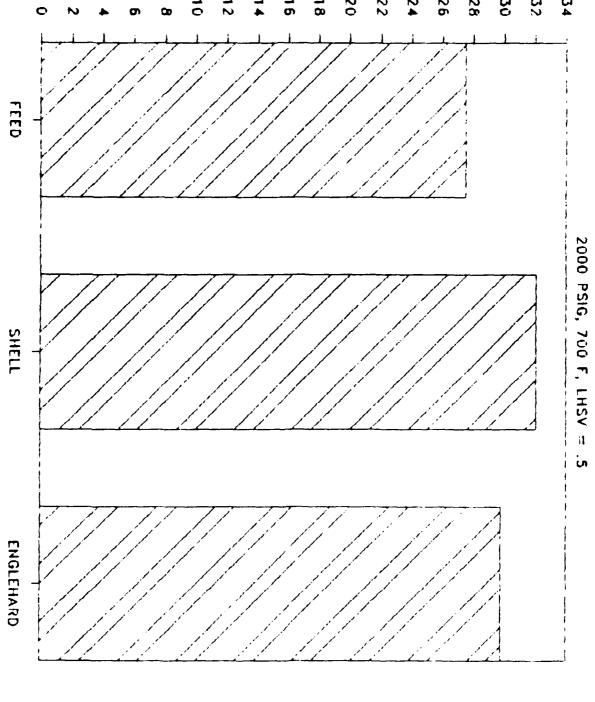
## EFFECT OF CALALYST TYPE ON MITROGEN



### WEIGHT \* HYDROGEN

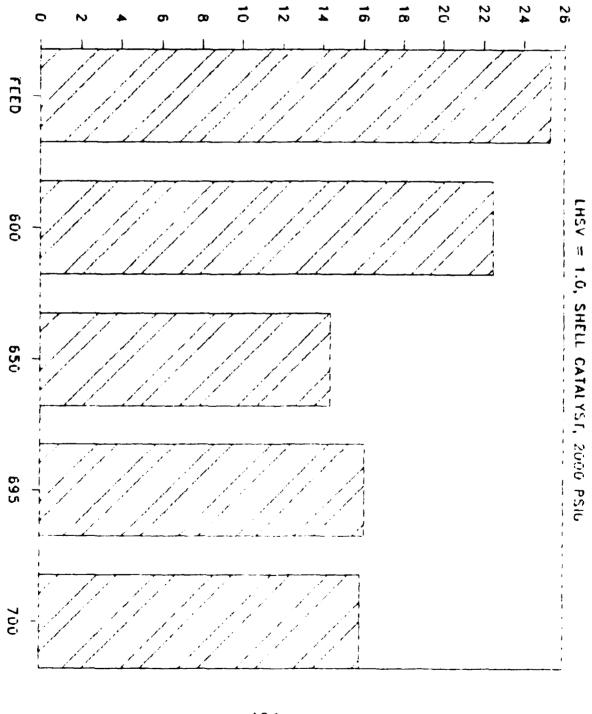


EFFECT OF CATALYST TYPE ON HIDROGED



EFFECT OF CAFALIST TYPE ON DENSITY

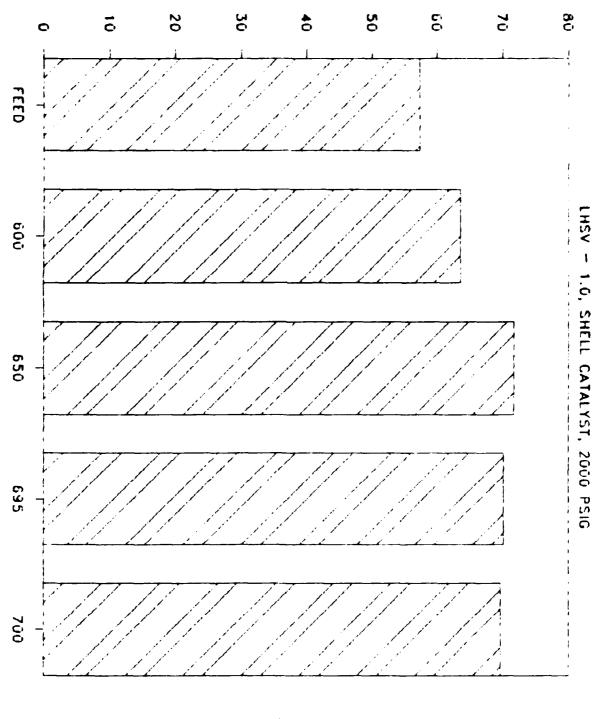
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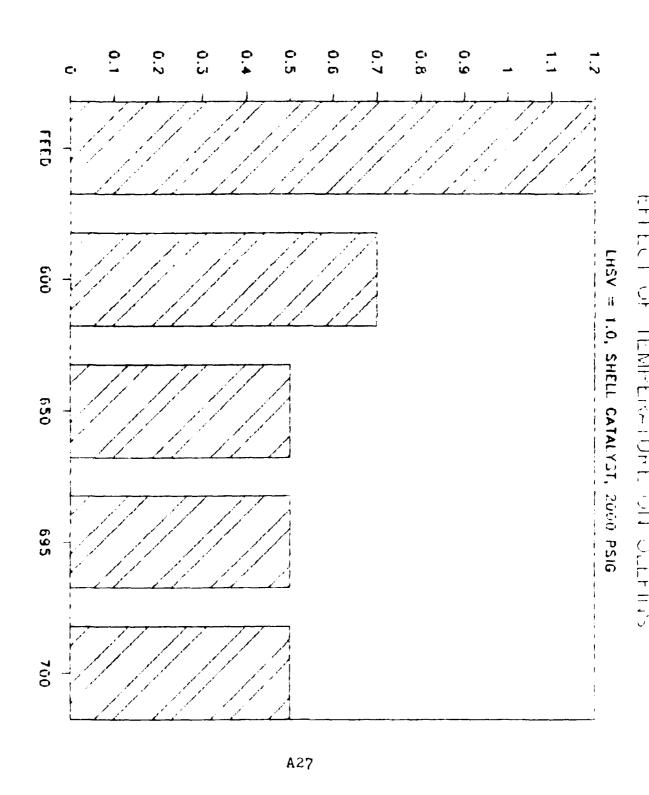
DIFFECT OF TEMPLEARIORS ON APOLICION

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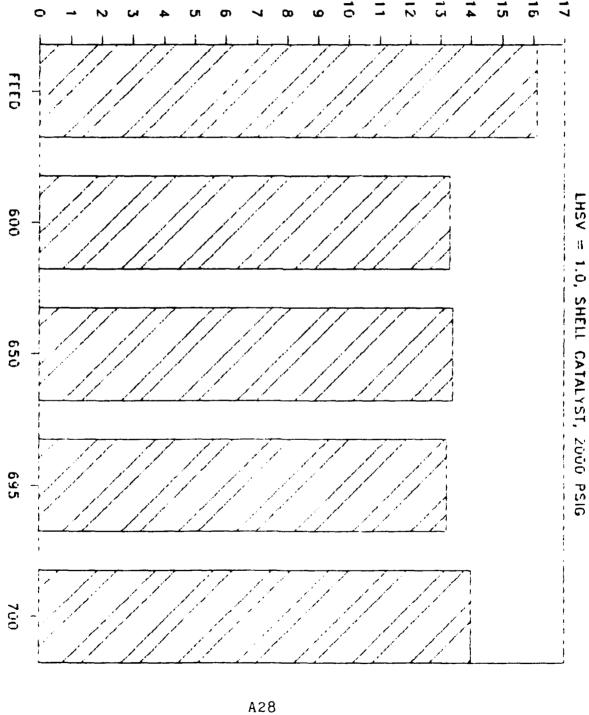
# EFFECT OF TEMPERATURE ON HAPHTHEMES



### WEIGHT & OLEFINS



### WEIGHT % PARAFFINS



EFFECT OF TEMPERATURE ON PARAFFITIS

[N] TOTAL SATURATES 20 びご 60 7ū 80 0 LHSV = 1.0, SHELL CATALYST, 2000 PSIG βŨŨ [ ] TOTAL UNSATURATES 650 695 7ūū A29

TEMP YOU GATORATED AND CHARLESTEE

### APPENDIX B



CATALYTIC, INC., P. O. Box 239, Wilsonville, AL (205) 669-6747

3 October 1986

Carl Smits J & A Associates 18200 West Highway 72 Golden, CO 80401

Dear Mr. Smits:

Attached is analysis of subbituminous solvent shipped to you on 23 September 1986.

Assistant Plant Manager

### WRH:mp

J. R. Gough

T. W. Johnson H. D. Schindler

J. Downer

Subbituminous coal feedstock was from Wyodak Upper Note: Seam, produced from Kerr-McGee mine near Gillete, WY.

The liquid was produced on Run #251 of the Advanced Coal Liquefaction Facility.

### Elemental Analysis:

wt 8 С 87.19 Н 12.40 N **0.23** S 0.01 H20 0.05 Sp. Gravity 0.8862

BTU Content = 20394.29 BTU/1b (Calculated using Elemental Analysis Number)

### GC SIMULATED DISTILLATION

V160 SN65975 INTEGRATOR II

SAMPLE DATE :9-18-65 GC RUN DATE :9-18-86 SAMPLE TIME : 1615

B2

INITIALS : KJ

LIGHT D	IL BOILING	FRACTIONS
---------	------------	-----------

LIGHT OIL BOILING	FRACTIONS	HEAVY OIL BOILING FRACTIONS	
TEMPERATURE  18P - 200 F  200 - 250 F  250 - 300 F  300 - 350 F  350 - 450 F  450 - EF F	WT % 6.46 7.64 5.54 6.35 12.48 61.52	TEMPERATURE WT % IBP - 350 F 26.00 350 - 450 F 12.48 450 - 500 F 10.54 500 - 550 F 9.98 550 - 650 F 20.02 650 - 850 F 18.94 850 - EP F 2.04 RESIDUE 0.	

IBE	=	97.0	F	100	_	<b>97.</b> 0	_
EF.	=	937.4	E	_			
		/ = / . 4	,	EF:	=	937.4	F

COMPONENT	BP F	WT %	CUM %	RT	KF
UNKNOWN	87.9	22			
PENTANE		.22	.22	72	870
UNKNOWN	97.0	.72	.93	78	865
LINE NOWN	133.4	.55	1.48	102	880
HEXANE	144.1	. 20	1.68	109	883
UNKNOWN	156.2	.80	2.48	117	878
	164.8	. 58	3.06	137	891
CYCLOHEXANE	176.0	3.16	6.22	163	735
UNKNOWN	200.0	.24	6.46	184	904
N-HEPTANE	209.1	.51	6.97	192	704
UNKNOWN	223.9	.02	6.99	247	
UNKNOWN	227.9	. 48	7 <b>.47</b>	262	918
TOLUENE	231.1	. 21	7.69		920
UNKNOWN	235.3	.77		274	797
FYRIDENE	240.8	4.31	8.46	286	925
UNKNOWN	242.6		12.76	302	917
UNKNOWN		. 84	13.60	306	928
UNKNOWN	248.4	. 49	14.10	319	930
F&M-XYLENE	266.3	2.68	16.78	359	935
UNKNOWN	281.1	.67	17.45	392	931
O-XYLENE	283.9	. 16	17.61	404	940
UNK NOWN	291.0	1.40	19.02	435	930
CHA PACMIN	299.1	.63	19.64	463	945
	- · ·				773

				. •	
UNI (NOWN	304.B	.09	19.90	483	947
UNKNOWN	308.6	1.68	21.58	496	747 948
UNKNOWN	319.5	1.03	22.61	534	951
FINKNOMN	333.6	2.78	25.39	583	954
NNKNOMN DIAKIADAM	341.9	.61	26.00	612	957
INDAN	3 <b>5</b> 0.0	1.10	27.10	640	797
- · -	354.4	1.11	28.21	658	960
UNKNOWN	359.6	. 1 1	28.32	679	1059
FHENOL	360.3	1.13	29.45	681	962
UNKNOWN	365.2	.47	29.92	695	762 963
UNKNOWN	37 <b>6.5</b>	1.67	31.59	727	766 966
UNKNOWN		.32	31.90	761	766 969
UNKNOWN	388.5	.32		7 <b>6</b> 2	
LINE NOWN	388.8 384.9	1.16	32.19 33.3 <b>5</b>	783	769 071
LINKNOWN	39 <b>6.</b> 2	1.68	35.03		971
TETFALIN	405.0		3 <b>5.4</b> 7	808	1022
NAPHTHALENE	424.0	. 44		840	1001
UNKNOWN	428.9	1.21	36.68 37.79	859	980 887
UNKNOWN	434.6	1.12		881	983 885
UNKNOWN	444.7	. 68	38.48	920	985
UNKNOWN	450.9	1.79	40.27	944	993
OUINOLINE	460.0	.31	40.58	979	1116
2-METHYLNAPHTHALENE	465.8	1.74	42.32	984	970
UNKNOWN	473.8	2.01	44.33	1014	1006
UNKNOWN	481.6	.80	45.13	1043	1013
UNKNOWN	485.0	. 68	45.81	1056	1015
BIPHENYL	492.0	1.51	47.32	1082	979
DIPHENYL-ETHER	496.3	1.70	49.02	1104	973
UNKNOWN	501.9	.63	49.65	1120	1031
UNK NOWN	511.3	1.63	51.28	1147	1039
UNF'NOMN	517.2	2.56	53.84	1164	1044
ACENAPHTHENE	531.1	. 75	54.59	1204	1114
UNENOWN	536.0	1.90	56.49	1217	1060
UNKNOWN	541.0	. 78	57.27	1230	1064
DIBENZOFURAN	548.6	1.72	58.99	1250	1123
UNF.NOWN	560.7	1.83	60.83	1280	1081
FLOURENE	568.4	1.53	<b>62.35</b>	1299	1137
UNF.NOWN	577.3	3.52	<b>65.</b> 87	1325	1098
UMF NOWN	586.3	. 88	66.76	1351	1108
UNF NOWN	593.8	.52	67.27	1373	1117
UNKNOWN	598.0	2.06	69.3 <del>3</del>	1385	1122
UNKINOWN	611.0	1.72	71.05	1423	1139
UNECNOWN	615.2	1.58	72.63	1435	1144
UNENOWN	620.3	1.54	74.17	1450	1151
UNKINOWN	633.4	2.52	76.69	1488	1169
FHENANTHRENE	643.0	1.67	78.36	1516	1187
UNENOWN	647.0	. 65	79.02	1529	1189
UNF NOWN	650.1	1.70	80.72	1539	1194
UMF NOMN	661.5	1.00	81.72	1576	1212
LNF NOWN	665.8	.60	82.31	1590	1220
UNF NOWN	670.B	1.60	83.91	1606	1228
1-METHYLPHENANTHREN		1.25	85.16	1623	1358
UNF,NOWN	680.7	. 75	85.91	1640	1246
9-METHYLANTHRACENE	685.4	.46	86.38	1657	1236
UNK NOWN	687.8	.63	87.01	1669	1262
UNKNOWN	697.9	2.18	89.18	1691	1274
UNF NOWN	711.6	1.17	90.35	1728	1295
FLOURANTHENE	721.9	.79	91.15	1756	1295
UNKNOWN	727.0	1.09	92.24	1771	1320
UNKNOWN	732 <b>.5</b>	.49	92.73	1787	1329
FYRENE	740.0	.91	93.64	1809	1365
UNENDWN	755.6	1.13	94.77	1850	1366
UNKNOWN	761.3	.30	95.07	1865	1374
NMCNEMU	767.0	. 19	95.27	1880	1383
UNF NOWN	776.5	.43	95.69	1905	1398
UNIF NOWN	783.7	.84	96.54	1924	1409
takir birashi	702.7	. 07	\ \	1000	

CONTRACTOR	<b>838.</b> 0	.71	7/.73	2067	1336
LINKNOWN	849.9	.03	97.96	2116	1517
UNKNOWN	857.4	.33	98.29	2147	1532
UNKNOHN	857.6	. 28	98.57	2148	1533
UNKNOWN	870.2	.07	98.64	2200	1558
UNKNOWN	879.7	. 43	99.07	2239	1576
UNKNOWN	906.1	. 37	99.44	2348	1618
PERYLENE	937.4	. 24	99.68	2477	1651
UNKNOWN	975.0	.17	99.85	2632	1678
UNKNOWN	1020.3	. 09	99.94	2819	1759
UNKNOWN	1074.9	.06	100.00	3044	1855



CATALYTIC, INC., P. O. Box 239, Wilsonville, AL 35186 (205) 669-6747

### 15 December 1986

Mr. John Downer 4597 Jupiter Drive Salt Lake City, Utah 84124

Dear Mr. Downer:

Enclosed are the run conditions and schematic sample location for the sample of solvent sent to you on 23 September 1986. The solvent you received was generated during Run 251-IID period. The solvent was from the low pressure flash (10-15 psig) of the 2nd stage reactor effluent.

If I can be of further assistance please advise.

Sincerely,

William R. Hollenack Assistant Plant Manager

WRH:mp

cc: J. R. Gough w/out encl.

H. L. Crean w/out encl.

G. A. Styles w/out encl.

T. W. Johnson w/out encl.

### Overall TSL Yields (Phase 3 Data)

	251-II#	251-IIC	251-IID
Operating Period	8/2-8/5	8/7-8/11	8/14-8/18
Date, 1986	8/2.8/3	8/7,8/8	8/14.8/15
Days selected	8/4.8/5	8/10.8/11	8/17,8/18
		354	249
Coal feed rate, MF lb/hr	353	334 33. 1	33.3
Coel conc. in slurry, wt & MP	33.0	33.1	33.3
Process solvent, wt 1		26(34)	26134)
Resid (A)	25(33)	24	24
cı	24	24	••
1st stage		**=	967
Reaction temp., of (average)	819	818	= -
Inlet H2 part. press., psia	2,510	2,530	2,530
Coal space rate,	_		16.8
MP 1b/hr/ft <sup>3</sup> (>700°F)	23.7	23.9	1.5
Fe <sub>2</sub> 0 <sub>3</sub> addition, wt % MF coal	0.8	1.5	1.2
2nd stage			
Reaction temp., *F (average)	743	745	745
Inlet Ho part, press, psia	2,550	2,560	2.500
Space velocity, hr 1	2.79	2.79	1.99
Catalyst type	Amocat 1C	Amocat 1C	Amocat 1C
Catalyst age, 1b (resid+CI)/1b cat	915-1028	1105~1254	1339-1443
CSD			
DAS type	2204	2204 - 2254	2254
H2 consumption, wt & MAP	6.3±0.1	6.020.1	6.3±0.1
Energy rejection, &	12.720.7	13.721.2	12.6±0.6
Yield, wt & MAF coal			14.1±0.5
Water	13.6±1.1	13.0±0.7	8.2±0.5
H <sub>2</sub> S, CO, CO <sub>2</sub> , WH <sub>3</sub>	10.1±0.4	10.420.8	6.0±0.8
C1-C3 948	8.1±0.3	7.020.4	60.7±1.8
C4+ distillate	61.0±1.3	58 - 42 1 - 6	10.9±1.1
C <sub>4</sub> + naphtha	19.7±1.1	19.3±1.4	11.0±0.4
Middle distillate	10.3±0.6	11.4±0.7	30.0±3.0
Distillate solvent	31.1x1.9	27.6±0.9 7.3±1.8	7.9:1.7
Resid (b)	3.922.2	7.3g1.g 9.3g1.0	8.7±0.3
Ash concentrate	9.4±0.#	9.321.0	φ. / <b>ξ</b> υ. 3
H2 efficiency			
1b C4+ dist/1b #2 cons	9.7±0 1	9.7±0.3	9.7±0.3
C1-C1 selectivity (X100)			
to C4+ distillate	13.0±1.0	12.0±1.0	10.0±2.0
Coal conversion, wt & MAP (c)		_	
1st stage	94.220.8	94.7±1.1	94.421.0
1st and 2nd stage	95.4±0.8	95.7±0.4	96.020.3
Two stage	95.4:1.3	95 · 3±0 · 4	94.7±0.7
(Resid + UC) conversion,			
wt & feed (d)			
ist stage (e)	34.821.1(61.021.8)	35.621.7(62.323.1)	37.0g1.4(64.1g1.8)
2nd stage	23.4±1.3(27.2±1.4)	17.82.1(20.1:2.7)	18.421.7(20.2:2.3)

<sup>(</sup>a) Data in parentheses on CI-free basis.
(b) Includes TSL system UC accumulation.
(c) Cresol solubles.

<sup>(</sup>d) Data in parentheses are based on MAP coal.
(e) MAP coal as 100 or % UC.

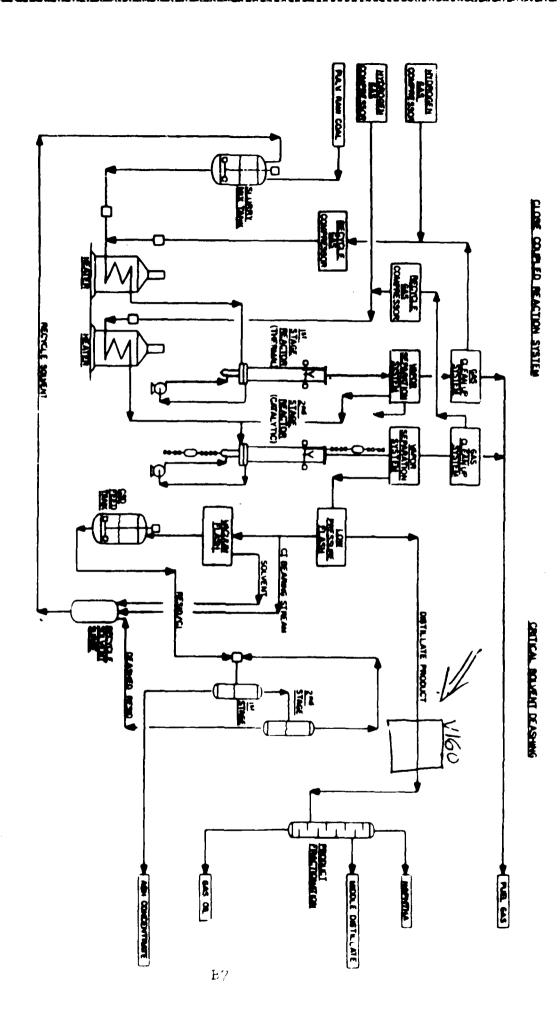
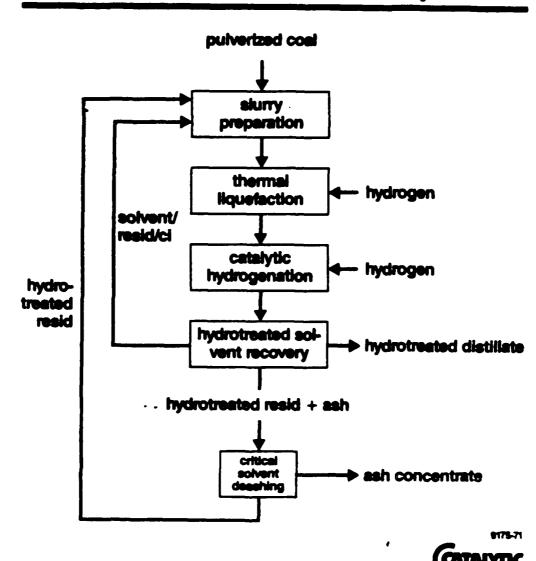


FIGURE 3. CLOSE COUPLED INTEGRATED TWO STAGE LIQUEFACTION SYSTEM
THERMAL - CATALYTIC WITH CLIRECYCLE

### CC-ITSL with ash recycle two-stage liquefaction close-coupled mode





### J&A ASSOCIATES, INC.

18200 West Highway 72 Golden, Colorado 80401

(303) 425-6021

October 27, 1986

Mr. John Downen

Subject: Preparation of Coal Liquid

AFWAL/POSF .

Hydrotreater Feed

Wright Patterson Air Force Base Reference: J&A Data Report #

OHIO 45433-6563

86-10-582-3510

Dear John,

As you requested in your P. O. # WL1, we have distilled two drums of Subbituminous Coal Liquid to your specifications in order to prepare a feedstock for hydrotreating tests. The drums were received from Catalytic, Inc. Wilsonville, Alabama on August 29, 1986 and the desired distillate was shipped to Wright-Patterson AFB on September 15, 1986. The feedstock produced consisted of 61.3 gallons of 26.6 API oil.

The distillation equipment used was our 25 gallon Batch Still (Figure 3) which was run at atmospheric pressure until the kettle reached 550°F and then under vacuum to take the 625°F cut (approximately 500°F max kettle temperature). This still takes cuts similar to those obtained from a True Boiling Point (TBP) distillation, ASTM D 2892. Each run of 25 gallons took two (2) days, even using a reflux ratio of 1:1 in order to speed up the work. During the distillations, data were recorded each time a receiver was drained (3.5 liter). Products were nitrogen blanketed as they were collected in order to preclude oxidation and polymerization.

Results are given in the enclosed tables and figures.

Table 1 gives the overall results and the analyses requested on the 300-625°F fraction (hydrotreater feedstock).

Tables 2 - 5 are the distillation reports giving weights, volumes, recoveries, and readings from the four (4) runs performed.

Figure 1 is a composite distillation graph, showing points from all four (4) runs.

Figure 2 is a plot of the product (300-625°F) density versus temperature, as requested by Captain William Harrison III.

Results obtained agreed reasonably well with the Catalytic, Inc. simulated distillation which predicted a 55.5 weight percent yield of 300-625 F, versus our result of 59.0 weight percent. Our initial D86 distillation indicated a 53% yield, but this distillation was run very slowly due to intense foaming of the sample. Therefore the D86 result on the whole oil is not reported here and should be discarded if you have a copy.



### Page 2

The IBP-300°F and 625°F+ fraction have been retained. Please advise us as to their disposal. Small samples of the whole oil and the 300-625°F fraction have also been retained if you need more analyses performed.

If you have any questions about the results or procedures, please call me or Dr. Mark Atwood at 303-425-6021.

Very truly yours,

( In Smits

CARL M. SMITS Laboratory Supervisor

CMS/eac

Encs.

cc: Mr. Dennis Morrell,

Hercules Res.

Mr. Timothy Dues,

WPAFB

Captain William Harrison III, WPAFB Dr. Mark T. Atwood, J&A A

J&A Associates

File Copy

Table 1

OVERALL RESULTS ON TWO DRUMS OF SUBBITUMINOUS COAL LIQUID

J&A Sample #	<b>#</b> 56503 <b>-</b> A	<u> </u>	<u> </u>	
TBP Fraction	IBP-300°F	300-625°F	625°F+	<u>Totals</u>
Weights, g	50,330	207,780	94,355	352,465
Wt \$ of charge	14.29	<b>58.</b> 98	26.78	100.05
Volumes, mls at 60°F	66,990	232,098	98, 178	397,266
Volumes, gallon at 60°F	17.7	61.3	<b>25.9</b>	104.9
Volume percent of charge	16.89	58.52	24.75	100.16
API Gravity, 60/60	56.9 <sup>8</sup>	26.6°	15.78	27.8°
Specific Gravity	0.7513	0.8952	0.9611	0.8882
Elemental Analysis, wt \$				
Carbon (PE 240C)		86.83		
Hydrogen (PE 240C)		12.84		
Nitrogen (Antek)		0.25		
Sulfur (XRF)		0.01 (13	1 wppm)	
Oxygen (by difference)		0.07		
D86 Distillation, OF at 1	atmos			
IBP/5 vol \$ over		340/360		
10/20 vol \$ over		382/409		
30/40		437/466		
50/60		486/506		
70/80		524/544		
90/95		566/581		
EP, OF/EP, Vol \$ over		590/98.5		
Residue		0.5		
Recovery		99.0		

TBP DISTILLATION REPORT

-	Disk: Form Doc: JAAO 10/15/86	Actual Ba								Vol	56503-C	56503-A 56503-B	Cut Number	Dist Type J&A Number Sample Des
-	Form JAA0014 86	Balance Data: Charge: Recovery: Loss + Hold % Recovery:	32.9	24.7	20.9	15. 3	11.6	7 +		l % Over	forals	IBP(72)-300 300-625	femp. Range F at 1 Atm.	1BP
<u>.</u> .		 	456 432	362 4 2	ມ ຜູ ພິລ ຄ	281	112	208	72 (IBP)	Overhead Temp.		0	F 166	2 Drums Sub
-		82,885 83,045 (160)								emp. of	22,380 83,045	12,075 48,590	Weight (gm)	Proj #
- -		gms. 93,318 gms. 93,555 gms. (237) wt \$ 100.									27.00	14.57 58.62	Er sa	1 of 4 3510 Coal Liguid
-		8 mls. 5 mls. nls. 25 Vol									100.19	14.57 73.19	M NC 8	Received
-		s. Charge Data: s. s. Rusiduum Dat	74.2 75.3	66.7 70.4	62.9	50. 20. 4≃	51.7	47.9	40.4	Vol % over	0.9612	0.7544	Specific Gravity	of 4 Analyst Date Liquid Received 9/29/86 from
-		 22 22 1 1 1 1	620 625	600 600 600 600	575	5 5 6 6 8	533	518	664 084	Overhead Temp.	15.7	56.1 26.5	09/00 IAV	C. M. Sm. October Catalytic,
- Andrews		82,885 .8882 API 22,380 .9612 API								Temp, OF	23, 283 93, 555	16,006 54.266 70.272	Volume	Smits er 2, 1986 e. Inc.
		I = 27.8°									24.95	17.15 58.15 75.30	Volume Percent	111
		1						F	3 <b>1</b> 2		100.25	17.15 75.30	M Volume Percent	
			· ·						2/2/				30336	

## TBP DISTILLATION REPORT

Dist Type TBP  J&A Number 56503  Sample Description 2 Drums
Dist Type TBP Dist # 2 of 4 Analyst C  J&A Number 56503 Proj # 3510 Date Q  Sample Description 2 Drums Subbituminous Coal Liquid Received 9/29/86 from Cata
Analyst C. M. Smits Date October 4, 1986 ceived 9/29/86 from Catalytic, Inc.

	33.1	29.8	26.6	23.3	20.1	16.7	13.2	10.0	6.7	\ \ \ \	0.2	Vol \$ Over	70707-0	れたのう。つ	56503-B	56503-A	Cut Number
	124	399	376	352	340	300	266	237	206	173	73 (IBP)	Overhead Temp, OF	Totals	Total Distilled	300-625	IBP(73)-300	Temp. Range F at 1 Atm.
													95,210	71,615	58, 110	13,505	Weight (gm)
													24.77	75.19	61.01	14.18	Wt 3
,	69.3	66.0	62.8	59.5	56.2	53.0	49.7	46.5	43.2	39.9	36.5	Vol \$ over	99.96	•	75.19	14.18	M Wt S
	587	574	563	545	532	520	512					Overhead Tem	0.9622		0.8973	0.7540	Specific Gravity
												D. OF	15.6		26.2	56.2	0 <u>06/60</u>
								•	77.1	75.8	72.5	mp. of Vol. 1 Over	24, <u>522</u> 107, 194	82,672	64.761	17.911	Volume (ml)
								(	625	617	601	Overhead Temp, OF	22.87 99.56	77.09	60.39	16.70	Volume Percent
										В1	13	υρ. ο <sub>Ε</sub>	99.96	-	77.09	-4 -5 -3 -5	Yoù ume Percent

NOTE: This distillation experienced a small vacuum leak. It is believed the recorded temperatures above 500°F should be approximately 10°F higher.

### Actual Balance Data:

\$ Recovery:	Loss + Hold-Up:	Charge:
99.96 Wt \$	72,410 gms. 107,194 mls.	95, 250 gms.
99.96 Vo	107.194 mls.	107,239 ml
¥ .	mls. Residuum Data: SG =	mls. Charge Data: SG =
SG	8 8	8
. 9622	.8982	1
API :	API :	
I = 15.60	= 27.8°	)

Disk: Form
Doc: JAA0014
10/15/86

## TBP DISTILLATION REPORT

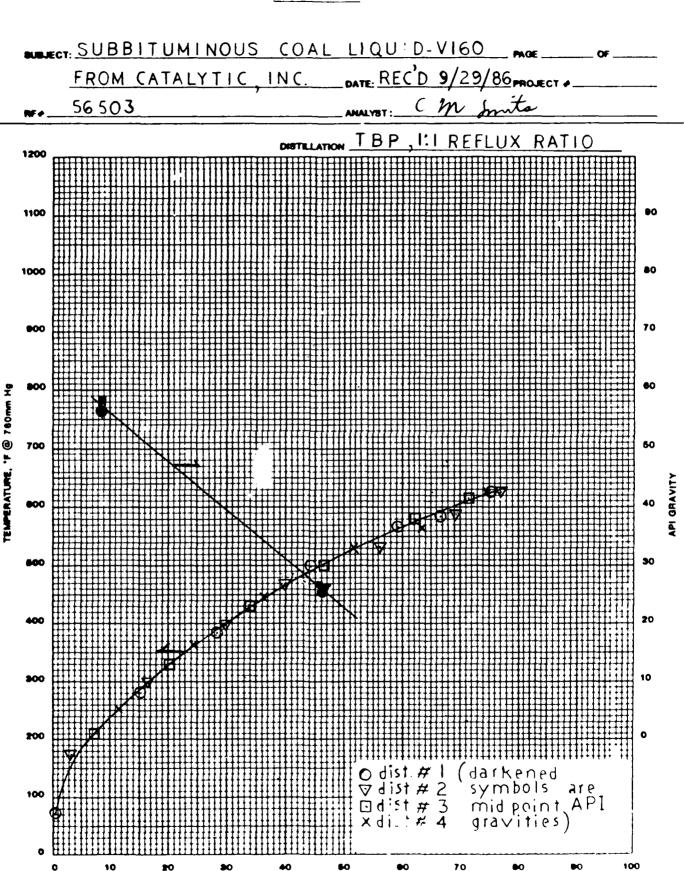
Actual Ba	329.5 ± 2 1 0 6 2 9 5	Vol \$ Over	56503-A 56503-B 56503-C	Dist Type J&A Number Sample Des Cut Number
Balance Data: Charge: Recovery: Loss + Hold-Up: % Recovery:	71 (IBP) 179 209 241 273 300 330 357 357 378 405	C Overhead Temp.	IBP(71)-300 300-625 Total Distilled 625+ Totals	pe TBP ber 56503  Description 2 Drums  Temp. Range F at 1 Atm.
92,880 92,990 (110) 100.12		, o	13,340 53,655 66,995 25,995 92,990	Dist # Proj # Subbituminous Weight (gm)
8ms. 104 8ms. 104 Wt 3			14.36 57.77 72.13 27.99	3 of 3510 Coal Lig
104,571 mls. 104,969 mls. (398) mls. 100.38 Vol	55.0 55.0 55.0 55.0 55.0 55.0 55.0 55.0	Vol % over	14.36 72.13 100.12	
mls. Charge Data: mls. mls. Residuum Dat	550 550 550 550 550 550 550 550 550 550	r Overhead Tem	0.7460 0.8940 0.9603	Analyst Date Received 9/29/86 from Specific Gravity
SG		P. of	58.2 26.8 15.9	Cal
92,880 .8882 .9603	71.5 74.5	Vol. % Over	17,882 60,017 77,899 27,070 104,969	October 9, 1986 alytic, Inc.  API Volume 0/60 (ml)
API = 27.8° API = 15.9°	615 625	Overhead Temp, OF	17.10 57.39 74.49 25.89	Volume Percent
1	B14	PP °F	17.10 74.49 100.38	Yolume Percent

Disk: Form
Doc: JAA0014
10/15/86

## TBP DISTILLATION REPORT

Actual Bal	31.9 31.9	Vol % Over 0.5 4.2 7.9 11.7	56503-A 56503-B 56503-C	Dist Type J&A Number Sample Des Cut Number
Balance Data: Charge: Recovery: Loss + Hold-Up: Recovery:	290 300 362 421	Overhead Temp. 83 (IBP) 181 215	IBP(83)-300 300-625 Total Distilled 625+ Totals	TBP 56503 cription 2 Drums Temp. Range F at 1 Atm.
81,275 ga 81,220 ga 55 ga 99,93 W			11, 410 47, 425 58, 835 22, 385 81, 220	Dist # 4 Proj # 3 Subbituminous Coal Weight (gm) Wt
8ms. 91.505 8ms. 91.548 8ms. (43) Wt % 100	711.0 711.0 711.0	Vol. % over 40.5 44.3	14.04 58.35 72.39 27.54	4 of 4 3510 Coal Liquid
.05		er Ver	14.04 72.39 99.93	1d Received  No. 10 Mt. 3
mls. Charge Data: mls. mls. Residuum Data Vol %	530 552 564 609	Overhead Tem 445 480 495	0.7511 0.8939 0.9606	Analyst Date 9/29/86 from Specific Gravity
		ED. OF	56.9 26.8 15.8	C. M. S October Catalytic. OAPI 60/60
81,275 .8882 API 22,385 .9606 API			15, 191 53, 054 68, 245 23, 303 91, 548	October 13, 1986  alytic, Inc.  API Volume 0/60 (ml)
$API = \frac{27.8^{\circ}}{15.8^{\circ}}$			16.60 57.98 74.58 25.47	Volume Percent
1		B15	16.60 74.58 100.05	Yolume Percent

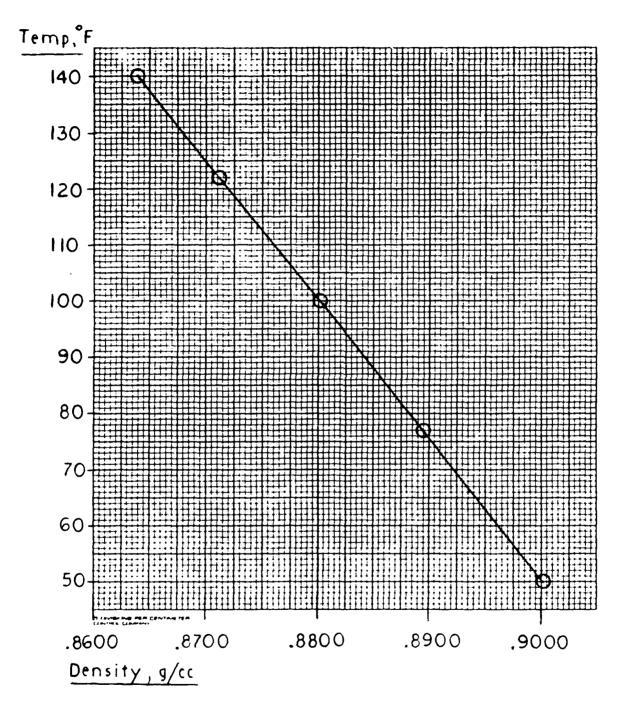
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VOLUME PERCENT DISTILLED

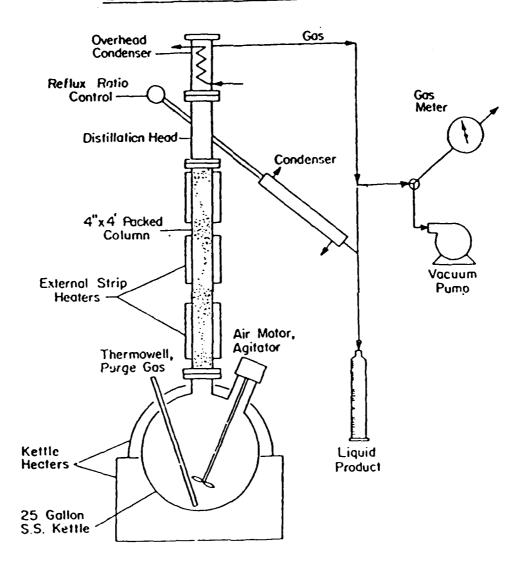
Figure 2

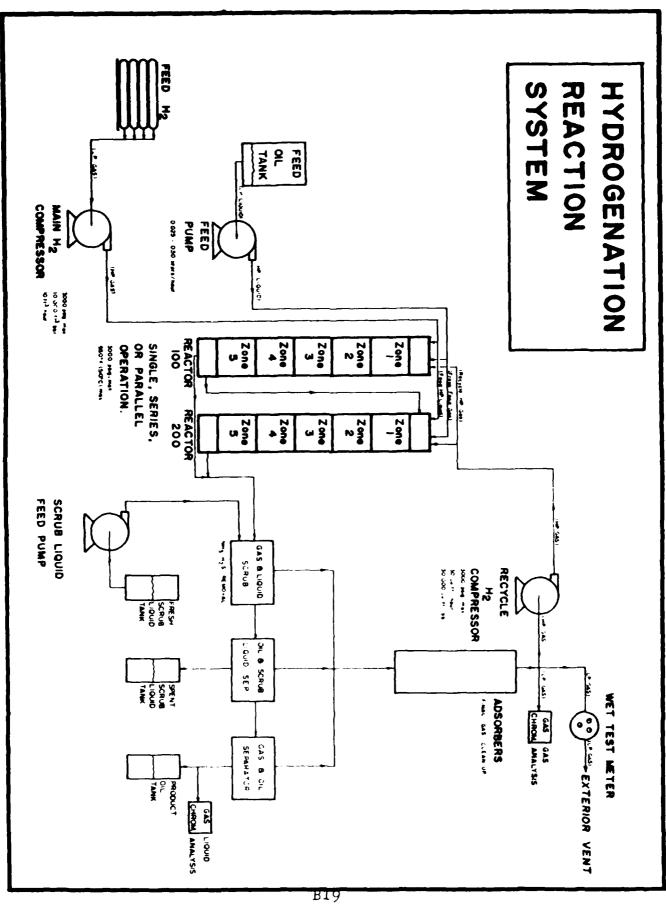
DENSITY VERSUS TEMPERATURE, 300-625°F FRACTION SUBBITUMINOUS COAL LIQUID



$\frac{\circ}{F}$	<u>°c</u>	Density, g/cc
50	10	0.9002
77	25	0.8895
100	37.8	0.8802
122	50	0.8712
140	60 B17	0.8639

FIG. 3
25 GALLON BATCH STILL





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# Shell 424 Ni/Mo Hydrotreating Catalyst

#### Introduction

Shell 424 is a third generation nickel molybdenum catalyst on an extruded alumina base. It was developed for use in severe, heavy oil services in which diffusional limitations may be a problem. This applies to all heavier than distillate feedstocks. Shell 424 provides much better activity and stability than traditional Ni-Mo catalysts. This catalyst differs from Shell 324 in several ways.

 The most significant change is a totally new technology for distributing the metals on the catalyst. The technique provides a much more even dispersion of metals throughout the catalyst.

- The base has been modified to make the catalyst more tolerant to metals and contaminants
- Shell 424 was the first shaped catalyst Shell produced commercially. The tri-lobed shape also reduces diffusional limitations. This catalyst has the same high strength characteristics that all Shell catalysts share, and the product has been dense loaded in nearly every application to date. The tri-lobed shape also provides reduced pressure drop compared with the same sized cylinder.
- The metal content was optimized for heavier feedstocks

#### Typical properties

Shape	Tritobed	To lobed	Trailoteed
Normal size, in (mm)	1.16 (1.5)	1/10/62 50	18730.
Chemical composition, % wt			
Nickel	3.0	3 ()	11
Molybdenum	130	130	175 (
Physical properties			
Surface area, m-/q	162	16()	156
Pore volume, co. g	0.47	() 47	0.4.1
Side plate crush strength*, lb (kg)	24 (10.9)	37 (16.8)	47 (20.9)
Bulk crushing strength?, kg/cm	16	17	18
Attrition index <sup>3</sup>	99+	98+	98
Reactor loading density, lbs/ft* (kg/l)			
sock loaded	47-48	47-48	47 48
dense loaded	52-53	52-53	52 53
Compacted bulk density, lbs/ft (kg/l)	52 (0.83)	52 (0.83)	52 (0.83)
Loss of ignition at 900° F (482 C), % wt4	0.7	0.7	0.7

<sup>12 16</sup> inch long particles

Container weights SD = 300 lb inet BP = 1500 lb inet

Pressure applied to procedures 0.5%W fines 40 mesh.

MWI retained on 20 mesh screen after tumbling 1 hour at 40 rpm

<sup>4</sup>As manufactured

#### Commercial experience

VGO hydrotreating and hydrocracter pretreating Shell 424 has found immediate acceptance throughout the industry. In less than one year it has been purchased by a dozen companies in over twenty locations throughout North America. It has been extremely successful in both first-stage hydrocrackers and VGO hydrotreaters. The differences in the base have also allowed refiners to put a significant amount of residue into cat. cracker pretreaters and also to treat de-asphalted oils at relatively low pressures. Shell 424 has improved stability and activity in all of these applications.

#### Mild hydrocracking

An additional use of Shell 424 has been in mild hydrocracking services. Several cat. cracker pretreaters are being used with Shell 424 to produce 20-30% additional distillate from VGO. The distillate has cetane indeces between 40 and 45 depending on the feedstock and cracking severity. Since the cracking reaction is primarily de-alkylation of polyaromatics, the bottoms product has a very low polyaromatic content and makes a superior cat. cracker feed. Shell 424 gives this 20-30% conversion with very low production of C4 and lighter materials.

#### Distillate hydrotreating

Shell 424 is in service treating coker and cracked distillates. The high hydrogenation, denitrification activity will yield improvement in product quality at high severity operations.

#### Packing and availability

Shell 424 is available as 1/16, 1/10, and 1/8 inch extrudates packed at 300 lbs net weight in 55 gallon steel drums or 1500 lbs net weight in sling bins. Orders normally can be filled on short notice from inventories maintained at Shell's West Coast manufacturing plant. Small inventories are typically kept on the Gulf Coast for emergency or top up needs.

## Health, safety, and environmental precautions

Shell 424 Catalyst is made from chemicals which span a range from being practically nontoxic to being potential carcinogens. Full attention to these hazards, and to appropriate precautions and preventative measures is essential. Before ordering, testing or using these catalysts, available information on health, safety, and environmental hazards, precautions, and preventative measures must be obtained from your Shell Chemical Catalyst Sales Representative.

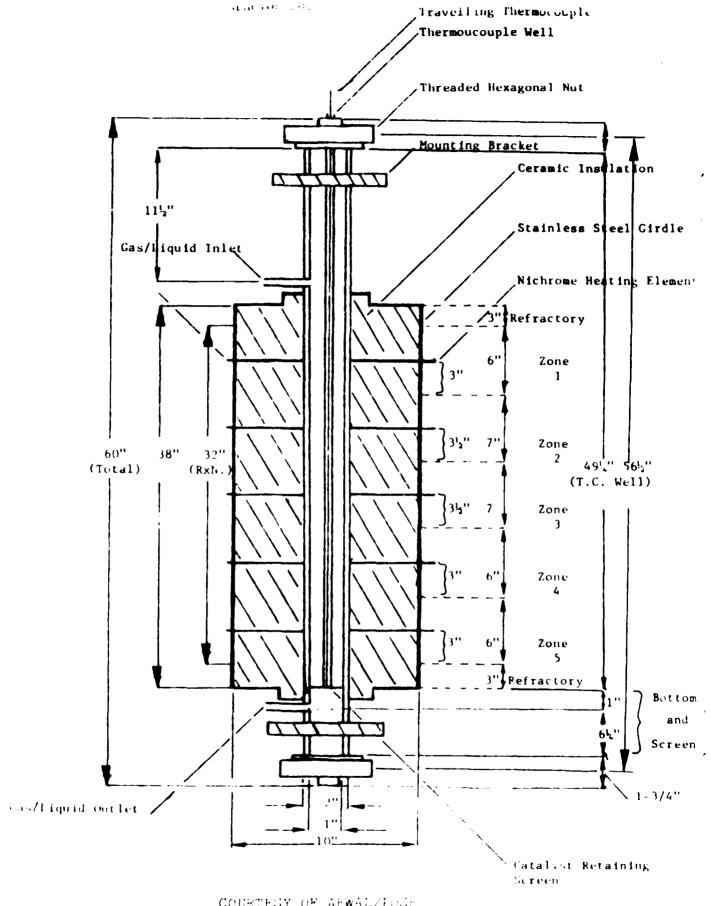
#### For additional information, call or write:

Shell Chemical Company Catalyst Business Center One Shell Plaza PO Box 2463 Houston, Texas 77252 Telephone (713) 241-4997 or 241-4927

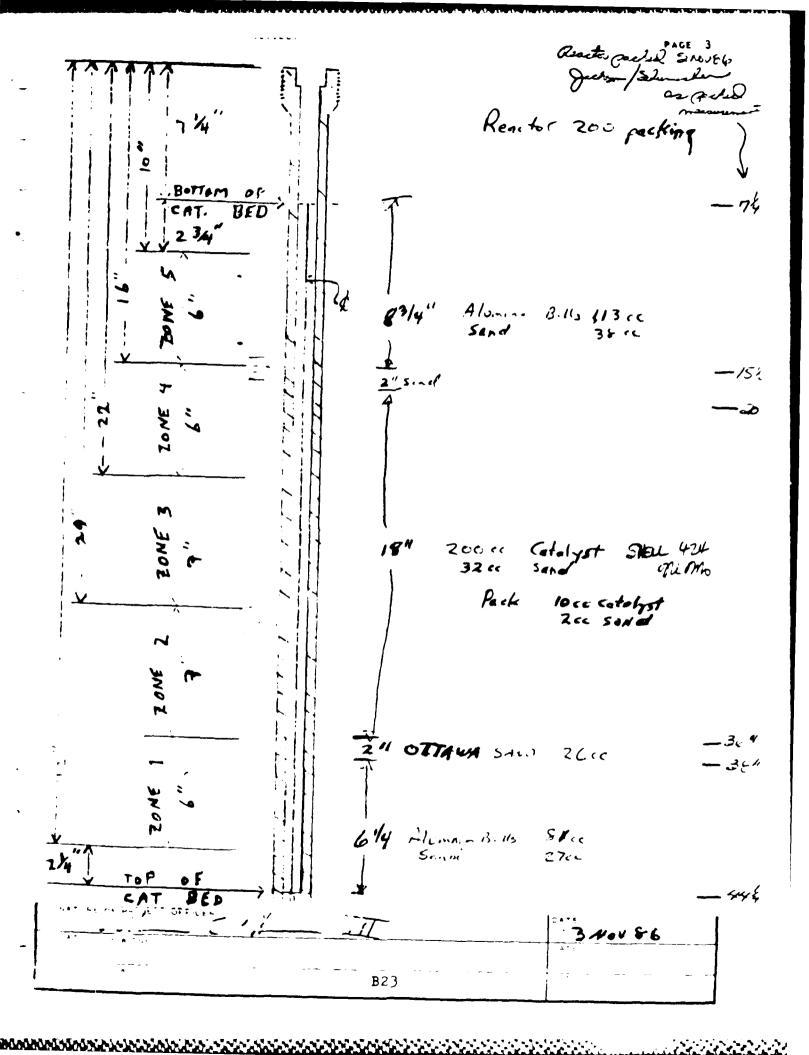
#### Warranty

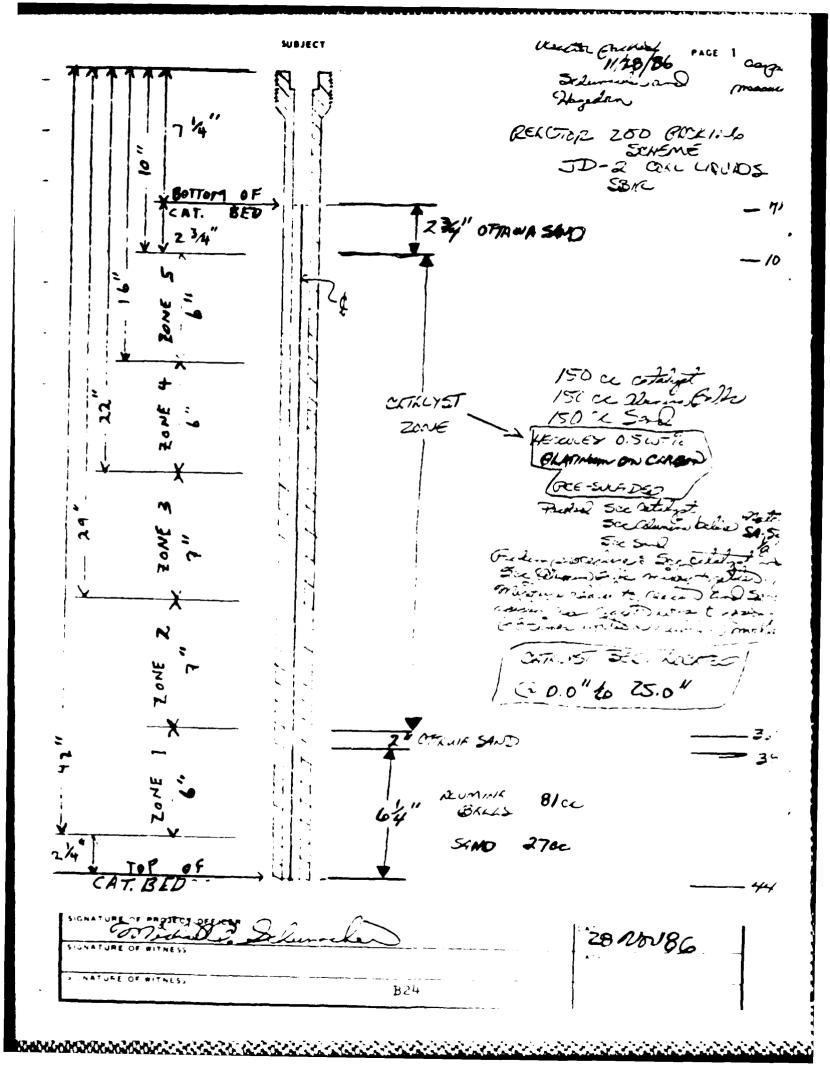
All products purchased from Shiril are subject to terms and conditions set out in the contract order acknowledgement and/or bill of lading. Shell warrants only that its product will meet those specifications designated as such herein or in other publications. All other information supplied by Shell is considered accurate but is furnished upon the express condition that the customer shall make its own assessment to determine the product's suitability for a particular purpose. No warranty is expressed or implied regarding such other information, the data upon which the same is based, or the results to be obtained from the use thereof; that any product shall be merchantable or fit for any particular purpose; or that the use of such other information or product will not infringe any patent.

Februar, 1985



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### APPENDIX C

John:

1. Here's a list of the test methods that were used to analyse the fuel samples for your program:

PONA ASTM-D-2789 (Pratt & Whitney) ASTM-D-2887 (SFTLA) Distillation Reichert ABBE MARK 11 (POSF) Ref. Index Total Aromatics Secific Gravity ASTM-D-1298 (SFTLA) NMR (POSF) Hydrogen Content Nitrogen Content Antec Nitrogen Analyser (POSF) Sulfur Content Horiba Sulfur Analyser (SFTLA) GC (POSF) GC/MS (POSF) ASTM-D-3338 (SFTLA) Net Heat of Comb. Thermal Stability ASTM-D-3241 (SFTLA)

- \* Reference "HYDROCARBON GROUP TYPE ANALYSER SYSTEM FOR THE RAPID DETERMINATION OF SATURATES, OLEFINS, AND AROMATICS ON HYDROCARBON DISTILLATE PRODUCTS" by Hayes and Anderson, Analytical Chemistry, November 1985.
- Indicates who did the actual analysis

Bob Morris

SH-HLO SETLA (J. YOUNT)

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C.Amili	•	

PLEASE PERSON THE FOLLOWING SPECIFICATION TESTS AND FORWARD THE SHIP TO CAPT. FILE HARRISON. AFMALL POSE, BLOG. 470, FORM 154, ENT. 58020. ANY QUESTIONS RESARDING THE PROSERTIES OF THIS SAMPLE CAN BE DIRECTED TO SHIP CAPTURE OF MIKE SCHUMACHER AT 58020. THAT YOU FOR YOUR SUPPORT AND COOSERATION.

EIGHER Robert W. Mans

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•	11RS-002	NR	0.1384	9.171	0.9464	NR
_	HRS-003	0.1998	NR	NB	0.9249	NR
1	FR5-004	1.6720	NR	NR	0.9563	NK
į	-RS-005	0.005301	NR	NR	0.9215	NR
٦	HRS-006	0.02297	0.1035	9,972	0.9279	NR
j	HRS-007	0.007144	0.1086	9.876	0.9236	NR
	-RS-008	0.003755	0.0469	12.379	0.3607	NR
٦	1285-009	0.03493	NR	7.136	1.0:52	NR
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